OPEN ACCESS
Saudi Journal of Oral and Dental Research
Abbreviated Key Title: Saudi J Oral Dent Res
ISSN 2518-1300 (Print) |ISSN 2518-1297 (Online)
Scholars Middle East Publishers, Dubai, United Arab Emirates
Journal homepage: http://scholarsmepub.com/sjodr/

Original Research Article

Evaluation of Surface Hardness and Surface Roughness of Different Nano Glass Ionomer Restorative Materials

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DOI:10.21276/sjodr.2019.4.8.7 | **Received:** 07.08.2019 | **Accepted:** 17.08.2019 | **Published:** 26.08.2019

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Abstract

This study aimed to compare the effect of simulated brushing on one Nano resin-coated glass ionomer (GI) (EF; Equia forte fil/Equia forte coat, GC Corp., Tokyo, Japan) and one Nano zirconia-reinforced GI (ZI; Zirconomer Improve, Shofu, Inc., Kyoto, Japan). One hundred and forty specimens were prepared and divided into two groups: EF and ZI. The Vickers Hardness Number (VHN) and surface roughness (Ra) were measured before and after simulated brushing of the specimens with water or with water and Colgate Total 12 (CT12) for 5,000, 10,000, and 20,000 cycles. Regardless of condition, EF and ZI showed statistically significant differences of mean VHN: 146.90 ± 36.66; 110.30 ± 13.73, (p-value < 0.001), and mean Ra: 0.290 ± 0.089µm; 0.247 ± 0.085µm (p-value ≤ 0.05), respectively. For both EF and ZI, repeated measures analysis of variance (ANOVA) revealed a statistically significant difference of mean VHN (p-value ≤ 0.05) in all cycles' intervals with CT12, but only at 20,000 cycles with water and in Ra (p-value ≤ 0.05) in all conditions compared to the control group. A post-hoc Tukey's test revealed a statistically significant difference of mean VHN in water vs. CT12 (p-value ≤ 0.001) for both EF and ZI in all conditions. The EF showed higher mean surface hardness and higher mean surface roughness as compared to the ZI. Both the EF and ZI specimens demonstrated resistance to the softening when being subjected to the simulated tooth brushing procedure equivalent to about 1 year of clinical use.

Keywords: Equia forte fil/Equia forte coat, Zirconomer Improve, Hardness Number (VHN), surface roughness, Nano glass ionomer, Simulated brushing.

INTRODUCTION

The synthetic biomaterials have been widely used to replace the damaged and lost tissues. It is considered as an old concept. There is a variety of restorative dental materials present in the field of dentistry. The silver dental amalgams are considered as one of the useful restorative materials. Dentists are using silver dental amalgams from almost about 150 years. With an advancement in dentistry, the glass ionomer cement (GIC) has been introduced as an important modern dental material. Glass ionomer cement (GIC) provides a revolutionary progression to the restorative approaches. Sidhu SK and Nicholson JW, Bonifácio CC *et al.*, and Francisconi LF *et al.*, In particular, it modernized the minimally invasive dentistry [1-3]. Wilson invented the glass ionomer cements (GICs) at the Laboratory of the Government Chemist in the early era of 1970s, the GICs are the water based cements. They are termed as the polyalkenoate cements. This is known as the generic name of the GICs on the basis of its chemical composition, an acid-base reaction taken place between the polyacrylic acid and the silicate glass. Bioactivity employed the biomaterial for performing the important procedures as, for inducting the cellular growth, for tissue formation and the proliferation. With addition to this, it also helps in enhancing the anti-bacterial effect of the dental material for the prevention or the treatment of the infection in the tissues. The significance of the glass ionomer cement (GIC) lies in the fact that it contains the alumino-fluorosilicate glasses that intrinsically contains the bioactive characteristics as it includes the fluorides and silicates. A large number of further modifications has been taken place in the glass ionomer cement (GIC) which produced the significant results or outcomes. With the addition of each modification, the obtained outcomes are considered or observed in depth relationship with its produced effects. It overall influences the concluding properties of the glass ionomer cement (GIC) [4, 5].

In the present day scenario, it is hard to consider any dental material as an ideal dental material with all the ideally acceptable dental applications. Like all other dental materials, glass ionomer cements (GICs) also have a number of flaws and drawbacks. GICs have many drawbacks such as, they consequently result in the fracture, can cause brittleness, its wear resistance is relatively poor, highly sensitive to the moisture on exposure to the oral cavity and inadequacy in the surface properties. Under these circumstances, the use of glass ionomer cements (GICs) have become limited or restricted for many clinical procedures Towler MR *et al.*, [6]. In this situation, a number of modern modifications have been introduced to the traditional glass ionomer cements (GICs) to overcome these shortcomings and to enhance the mechanical properties of the GICs Sidhu SK, Nicholson JW [1]. The most important of these modifications are the following. RMGICs are considered as the key modification to this GICs. RMGICs are termed as the resin-modified glass ionomer cements. RMGICs are formed as a result of the mixture of the glass ionomer cements (GICs) with the photo-curved or auto-curved resin systems. Other modifications are consisted of the following; fiber-reinforcement, the mixture of GICs with the polyvinyl phosphonic acid, zinc strontium oxide, silica particles, stainless steel, amino acids, zinc, N-vinylpyrrolidone and bioactive apatite with or without zirconia. The advance technology includes the Nano technology widely used the systems, the materials or modifications having the sizes ranging between 1 to 100 nm. Nanotechnology is considered vital in the area of dentistry with its incorporation of the Nano sized particles to modify the restorative dental materials and their applications in curing and preventing caries. According to the modern recent researches, the mechanical properties of the dental restorative materials like the resin composites can be enhanced by the incorporation of the “Nano clusters” or the Nano-sized particles. Researches are being conducted to evaluate the similar approaches to enhance the physical and mechanical traits of GIC by the help of nanotechnology.

Nanotechnology includes the two basic approaches for the manufacturing process of the Nano-sized particles. The two approaches are termed as, the top-down approach and the bottom-up approach. First approach named as top-down nanofabrication involves the elimination of the bulk material for the production of the Nano sized particles. The common examples of top-down nanofabrication are the machining, milling and the lithography. The second basic approach is termed as the bottom-up nanofabrication. It involves the formation of the Nano-sized particles atom by atom. The common examples that can explain the bottom-up nanofabrication approach are, the protein synthesis, tissue regeneration and the biomimetic dental implant coatings [7, 8]. In case of GICs, the Nano-sized particles production has been done mainly through the top-down nanofabrication approach.

This research has been conducted to evaluate the surface hardness and surface roughness of the different Nano glass ionomer restorative materials. For testing the surface hardness and surface roughness of the different Nano glass ionomer restorative materials, it is necessary to know about the different forms of modified Nano glass ionomers. The powder-modified Nano glass ionomers are also known as one of the forms of Nano glass ionomers [9]. The research of De Caluwe *et al.*, [10] concludes that the process of doping the Nano sized particles with the traditional or conventional glass ionomer cements (GICs) results in the enhancement of the compression strength and elastic modulus. At the same time, it also helps in reducing the setting time period of conventional GICs [10]. The process of thermo-cycling is widely used for the purpose of aging the restorative dental materials in the laboratory, artificially. It has been done to evaluate or assess the effect of the moisture and the oral temperature on the restorative materials. The response of thermo-cycling process is more deleterious on the mechanical characteristics of the Nano- filled glass ionomer cements on its comparison to the traditional glass ionomer cements. It concludes that, in order to enhance the various properties such as (physical, mechanical and biological) of the powder-liquid formulations of the glass ionomer cements, a number of Nano-sized powders can been mixed with the component of the glass powder. The two most famous modifications in this category are the modification using the Nano-Apatite and the modification with Nano-Sized Hap/Zr, CaF_2 and TiO_2 particles [11, 12].

Another important modification of glass ionomer cements (GICs) with the Nano-sized particles is known as the Nano-Filled Resin-modified glass ionomer cements. These modified GICs are rather complicated ones. They are different from the conventional GICs containing the glass powder and the polyacid solution. In addition to this, the resin modified glass ionomer cements contain the polymer resin component material which usually requires a self-activated or the light-activated polymerization chemical reactions to settled down. These are also known as the hybrid materials and aid in combining the mechanical characteristics of the resin composite with the anti-carious potential of the glass ionomer cements (GICs). The resin-modified GICs, known as RMGICs, are better than the conventional ones but they still carry some drawbacks. In order to reduce the drawbacks such as; the low level of fluoride release, brittleness and low strength, the RMGICs are attempted to combine with the Nano-sized fillers and the bio-ceramics particles. It produced the Nano-fillers resin-modified glass ionomer cements having the enhanced properties [13].

The Nano-particles are incorporated with the glass powder of the GICs which results in the size distribution of particles which brings about the higher modifications in the mechanical properties. They also help in filling up the empty spaces left between the glass ionomer particles. In this way, they perform as the reinforcing component in the formation of the GICs. Nano-filler element present in the Nano glass ionomers also aids in improving the physical characteristics of the hardened restorative materials. The micro-mechanical interlocking property in the chemical bonding procedure of Nano ionomers is mainly attributed by its surface roughness. In most general cases it also accompanies the chemical reactions by the acrylic or itaconic acid copolymers. The main advantages of the Nano glass ionomers are its advanced esthetics, improved polish and enhanced wear resistance.

The major focus of this research lies in testifying the surface hardness and surface roughness of the above explained Nano glass ionomer restorative materials. As the Nano glass ionomers are considered as the modified and improved version of the conventional glass ionomers and also considered as a best possible restorative dental materials, it is significant to conduct a research on its important properties. It enables the researcher to add in the advantages and usefulness of the Nano glass ionomers restorative materials.

MATERIALS AND METHODS

Materials

The conducted research mainly focuses on the Nano glass ionomer restorative materials. The study includes the different Nano glass ionomers. The product taken under experimentation includes; Equia forte Fil/ Equia forte coat GIC, Zirconomer Improved GIC and CT12 Clean Mint Dentifrice. The details and technical specifications of the material used for experimental procedure is explained in Table-1 as follows:

Table-1: Technical specifications of the tested Nano glass ionomer restorative materials, according to their manufacturers

Product Name	Composition	Manufacturer
Equia forte Fil/ Equia forte coat GIC	Equia forte Fil: highly reactive fluoro aluminosilicate micron-sized fillers, high molecular weight polyacrylic acid Equia forte coat: Cross-linking monomers and Nano fillers	GC Crop., Tokyo, Japan
Zirconomer Improved GIC	Fluoro aluminosilicate glass with ceramic and zirconia Nano fillers, high molecular weight polyacrylic acid	Shofu Inc., Kyoto, Japan
CT12 Clean Mint dentifrice	1450 ppm of fluoride, triclosan 0.3%, water, sorbitol, silicon dioxide (abrasive), sodium lauryl sulfate, PVM/MA copolymer (Grantrez), flavor, carrageenan, sodium saccharin, sodium hydroxide, white colorant	Colgate-Palmolive, São Bernardo do Campo, SP, Brazil

Method

This research uses the experimental approach for calculating the desired results to check the surface hardness and surface roughness of the different Nano glass ionomer restorative materials. The sample taken has been stated above.

Specimens' Preparation and Grouping

In order to conduct an experiment to determine the surface hardness and the surface roughness of the different Nano glass ionomers restorative dental materials, it is necessary to prepare the specimens for systematically for the required process. After being prepared properly, the specimens should be divided into the different groups.

The sample under consideration consists of one hundred and forty specimens. The specimens taken was first prepared for the procedure. After preparation, the respective specimens were divided into the two groups. Each group contains the 70 specimens. Basically, 70 specimens of each material has been taken and named as a one group. Similarly, the other group has been formed by taking the 70 specimens of the other material. For the preparation purpose of the specimens, a disk shaped split Teflon mold has been used. The split Teflon mold having a shape of disk is about 6mm in its diameter and 3mm in its height. The specimens were being prepared at the maintained temperature of 25±1°C. Then, the specimens were filled in the Teflon molds. It is important to take care that each of the Teflon mold should be slightly overfilled. After filling the molds, the molds were covered properly by the slide of glass microscope. The covering of molds by the slides helps in achieving the flat surface, smoothness and it also extrudes the excessive or extra material.

In next step, the taken specimens were covered properly with their respective protective coats, immediately. Then, the Nano coat of the EF specimens were cured by using the light-emitting diode curing unit. This was done for the time period of 20s. The manufacturer recommended to use the light emitting diode curing unit for 20s for this process. For ensuring the curing efficiency, the curing light of the light emitting diode was perpendicularly adjusted to the surface of the respective specimen at contact with the glass slide. The intensity of the light was adjusted at 600mW /cm^2. After this, the 70 under-experimentation specimens of the glass ionomers were divided randomly into four sub-groups. The subgroups are as: control (n=10) and 5,000, 10,000, or 20,000 cycles of the simulated tooth brushing (n=20). The three last subgroups were divided further. Each group of further divided into 2 sub-subgroups (n=10). After this further distribution, the next step implies the tooth brushing procedure differently for all three sub-subgroups. First, the sub-subgroup one was being exposed to the tooth brushing with the toothbrush using water only. Second, the sub-subgroup two was being expose to the tooth brushing with the toothbrush using the water along with CT12. Finally, all the specimens' under-consideration were stored and preserved in the distilled water at the temperature of 37^o C for the time duration of 24 hours before being exposed to any testing procedure.

Simulated Tooth Brushing Procedure

For performing a simulated tooth brushing procedure, it is necessary to select a suitable toothbrush simulator first. In this research, the toothbrush simulator (ZM-3.8; SD Mechatronic, GMBH, Germany) was used. This toothbrush simulator was selected because of its various properties. It is designed in such a way that it can imitate the various brushing movements on the specimens' surfaces with the assistance of the dentifrice. The toothbrush simulator was programmed to manage the complex motion sequences. The transverse backward and the forward motion along with the circular motion were programmed by using the simulator device to mimic the realistic brushing of the teeth. Another specification of this toothbrush simulator is that it can load up to eight specimens at the same time. The individual chambers are made for placing specimens, known as specimen chambers. These chambers are separated from one and other and are cleaned individually. The acrylic plates filled with the silicone impression materials are present for each specimen. Each specimen was fixed or placed in the center of each acrylic plate. The toothbrushes used for this procedure were the Medium-bristle Classic Colgate toothbrushes (Colgate-Palmolive, São Bernardo do Campo, SP, Brazil) were selected to use at 200gm load for 5,000, 10,000 and 20,000 cycles, respectively. According to the research study design, the tooth brushing procedure of the specimens was done by using either the wet toothbrush with the Colgate Total 12 tooth dentifrice or with the wet toothbrush without any dentifrice. Another important caution is the adjustment of the toothbrush holding apparatus. The toothbrush holding portion of the device was adjusted in a way that all the bristles were remain in constant contact with the specimen. Following the test, the specimens taken were removed and then rinsed with the distilled water for about 15 seconds, then air-dried.

Tests

For completing the followed procedure, the specimens are subjected to testing to determine its surface roughness and its surface hardness. Following are explained the relative tests performed in this research study;

Surface Roughness Testing

Surface roughness testing was performed to determine the level and changes of the surface roughness for the specimens' under-observation. For all the specimens, the surface roughness was evaluated by using an automated bench top profiler (Contour GT; Bruker, Billerica, MA). The specified profiler used has a capability of performing the 3D surface measurements of features ranging from 0.1 to 10mm in height. Each specimen was scanned on its surface at three sites one by one and the mean Ra (μm) was obtained. The profiler was calibrated after every three specimens by testing the surface roughness of a standard pretested specimen.

Surface Hardness Testing

The surface hardness of the specimens were evaluated by using the Vickers Hardness Test (VHT; Micromet 6049; Buehler, Illinois, USA). The test was used to perform the three indentations (load: 0.1 kg; dwell time: 5 seconds) on the upper top surface of the each one specimen in all the groups after being tested for the surface roughness. Then, the length of the diagonals (d1 and d2) that were left behind by the indenter were measured digitally (Stemi SV6 with Axiovision LE; Carl Zeiss Microscopy, Jena, Germany). After this, the mean value of all of these three measurements was calculated for each of the specimen. The VHT was calibrated after every three specimens by testing the surface hardness of a standard pretested specimens.

RESULTS AND DISCUSSION

After performing the procedure on the specimen under-observation, a number of different results have been evaluated. The results has been obtained by performing the tests on the processed specimens. The tests are performed for surface hardness and surface roughness. The results reveal the mean values and significant changes regarding different Nano glass ionomer restorative materials under different conditions.

Statistical Analysis

Statistical analysis of data obtained is necessary in order to understand and state the proper results. The obtained data was statistically analyzed by using the SPSS-20.0 (IBM product, Chicago- USA). Mean ± Standard Deviations were calculated to evaluate and state the numeric variables including the roughness and hardness based on the multiple readings on the baseline (control), at 5000, 10,000 and 20,000 cycles. The numerical data were explored for normality by checking the division of the data and using the tests of normality (Kolmogorov-Smirnov and Shapiro-Wilk tests) **(**Tables 7 & 8**)**. The analysis proposed that the data distribution is normal (parametric). In order to analyze the effect of restorative material, condition and their interaction on the mean surface hardness and the mean surface roughness, the Two- way Analysis of the Variance (ANOVA) was employed. In order to take a comparison between the k-related sample data sets in relation to the toothpaste and water effects within each restorative dental material, the repeated ANOVA tests was implemented to analyze the effect of water and CT on various cycles in comparison with the controlled group. Another important test named Post-Hoc Tukey's test was performed to do the comparative analysis of the effect produced by water in relation to the one produced by the toothpaste measured on various intervals. The P-value ≤ 0.05 was analyzed to be the statistically significant difference of means.

Tables 2 & 3 can explain the results obtained by performed tests;

Table-2: Surface roughness (µm) of the two different Nano glass ionomer restorative dental materials

Material	EF			ZI		
	Water (W)	Toothpaste (CT)	p-value	Water (W)	Toothpaste (CT)	p-value
Control	0.1273±0.0077	0.1273±0.0077	--	0.1211±0.0104	0.1211±0.0104	--
5000	0.2421±0.0049	0.3189±0.0173	0.000	0.2422±0.0414	0.2285±0.0167	0.316
10000	0.2434±0.0072	0.3424±0.0192	0.000	0.2231±0.0275	0.2389±0.0205	0.071
20000	0.3459±0.0413	0.4113±0.0167	0.000	0.2572±0.0356	0.4156±0.0198	0.000

The table shown represents the results obtained on testing the taken specimens of the different Nano glass ionomers restorative dental materials for checking its surface roughness. The two materials are taken and analyzed statistically. The table shows the statistical values obtained as a result of testing. It represents the significant difference in the mean values of the surface roughness under 5000, 10,000 and 20,000 cycles of the water and the toothpaste as compared to the control group in both the cases having two different dental restorative materials at the 5% level of significance.

Table-3: Surface hardness (VHN) of the two different Nano glass ionomer restorative dental material

Material	EF			ZI		
	Water (W)	Toothpaste (CT)	p-value	Water (W)	Toothpaste (CT)	p-value
Control	131.21±1.42	131.21±1.42	--	102.52±2.04	102.52±2.04	--
5000	132.44±2.99	168.28±2.89*	0.000	101.91±2.46	108.92±1.53*	0.000
10000	132.20±4.49	182.97±2.43*	0.000	101.92±0.93	116.07±2.71*	0.000
20000	78.90±1.41*	194.32±10.48*	0.000	99.91±2.04*	140.86±0.99*	0.000

The table shown above represents the results obtained by performing tests on the taken specimens of the different Nano glass ionomer restorative dental materials for evaluating its surface hardness. The two different Nano glass ionomer restorative dental materials were taken as a sample. The statistical analysis evaluates the statistically significant difference in the mean values of the hardness under the various cycles of the water and the CT on its comparison with the controlled group at 5% level of the significance **(**Tables 4 & 5).

Table-4: The table shows the numerical results for the Two-way ANOVA roughness test performed to evaluate the surface roughness of the two different Nano glass ionomer restorative dental materials

Roughness	Type III Sum of Squares	df	Mean Square	F	P-value	Partial Eta Squared	Observed Power
Material	0.073	1	0.073	7.138	0.228	0.877	0.166
Medium	0.347	1	0.347	34.190	0.108	0.972	0.354
Material *Medium (Interaction)	0.010	1	0.010	2.004	0.159	0.015	0.290

Table-5: The tabular representation shows the numerical results of the Two-way ANOVA hardness test performed to evaluate the surface hardness of the two different Nano glass ionomer restorative dental materials

Hardness	Type III Sum of Squares	df	Mean Square	F	P-value	Partial Eta Squared	Observed Power
Material	53533.793	1	53533.793	3.755	0.303	0.790	0.122
Medium	57009.275	1	57009.275	3.999	0.295	0.800	0.126
Material* Medium (Interaction)	14257.271	1	14257.271	56.248	0.000	0.293	1.000

Table-6: This table shows the effect of the material on the surface roughness and the surface hardness regardless of any condition

	EF		ZI		p-value
Measurement	Mean (SD)	95% CI	Mean (SD)	95% CI	
Roughness	0.290 (0.089)	0.269-0.311	0.247 (0.085)	0.226-0.267	0.004*
Hardness	146.90 (36.66)	138.16-155.64	110.30	107.03	<0.001*

*: *Significant at* $P \leq 0.05$

Table-7: Descriptive Statistics; One- Sample Kolmogorov-Smirnov Test

	N	Mean	Std. Deviation	Minimum	Maximum
Roughness	140	.26842	.089493	.095	.458
Hardness	140	128.602	33.1366	76.7	210.6

Table-8: One Sample Kolmogorov-Smirnov Test

		Roughness	Hardness
N		140	140
Normal Parameters[a,b]	Mean	.26842	128.602
	Standard Deviation	.089493	33.1366
Most Extreme Differences	Absolute	.105	.132
	Positive	.105	.132
	Negative	-.096	-.095
Kolmogorov-Smirnov Z		1.237	1.565
Asymp. Sig. (2-taileed)		.094	.015

Test distribution is Normal

a) Calculated from the data

The following figurative analysis **(**Figures 1-4**)** shows the condition of the EF and ZI specimens during testing procedure. A substantial difference has been observed before and after tooth brushing.

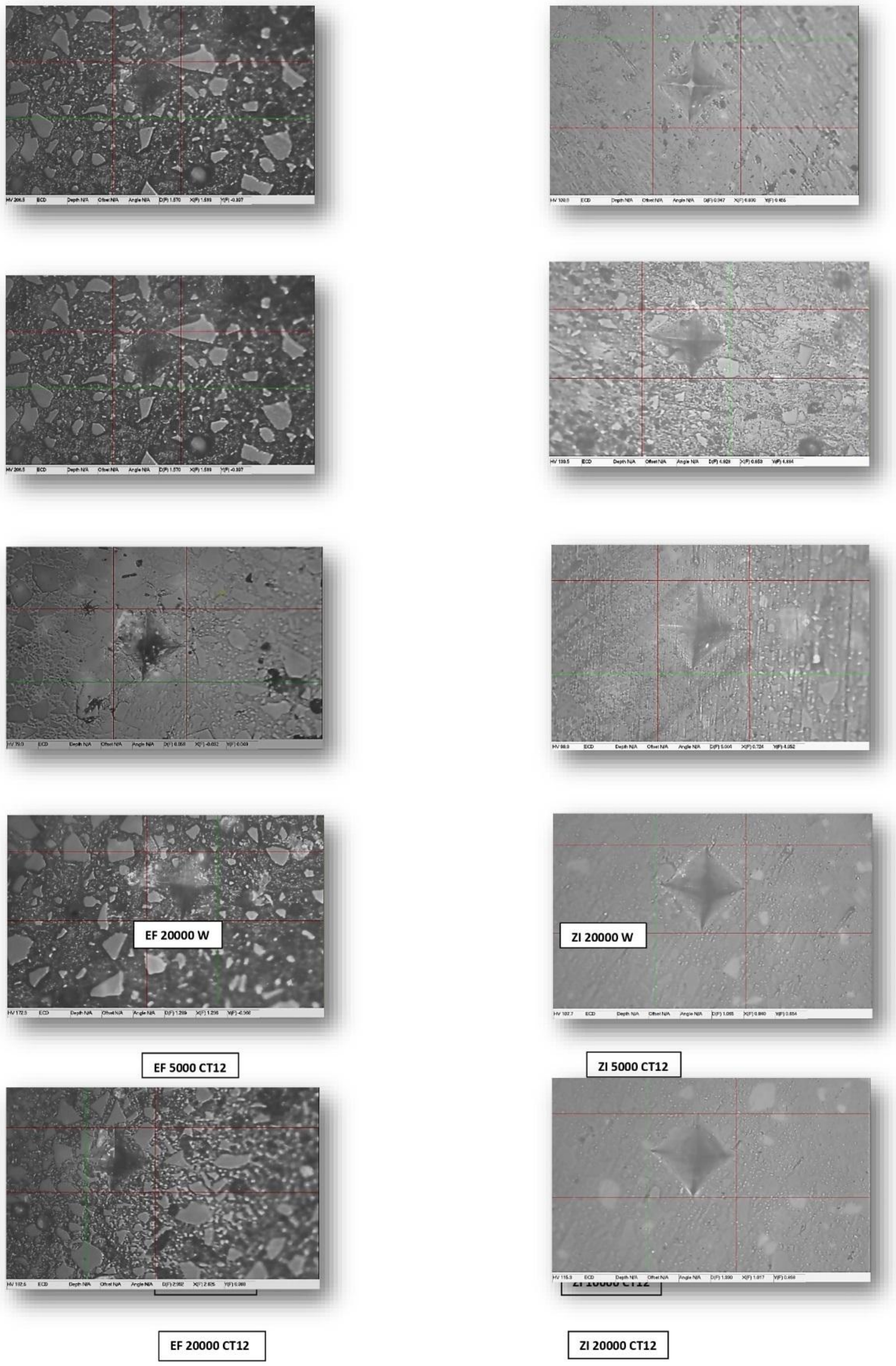

Fig-1: The above figures shows the hardness profile of the respective EF and ZI nano glass ionomers. Figure a; shows the EF specimen in control state, figure b; shows the ZI specimen in control state, figure c and d; shows the EF and ZI specimens after being subjected to simulated tooth brushing procedure 20,000 cycles with water wetted toothbrush only, figure e, f, g, h, i and j; shows the specimens of EF and ZI after being subjected to the simulated tooth brushing procedure for 5,000, 10,000 and 20,000 cycles while using toothbrush wet with water and tooth dentifrice (CT12)

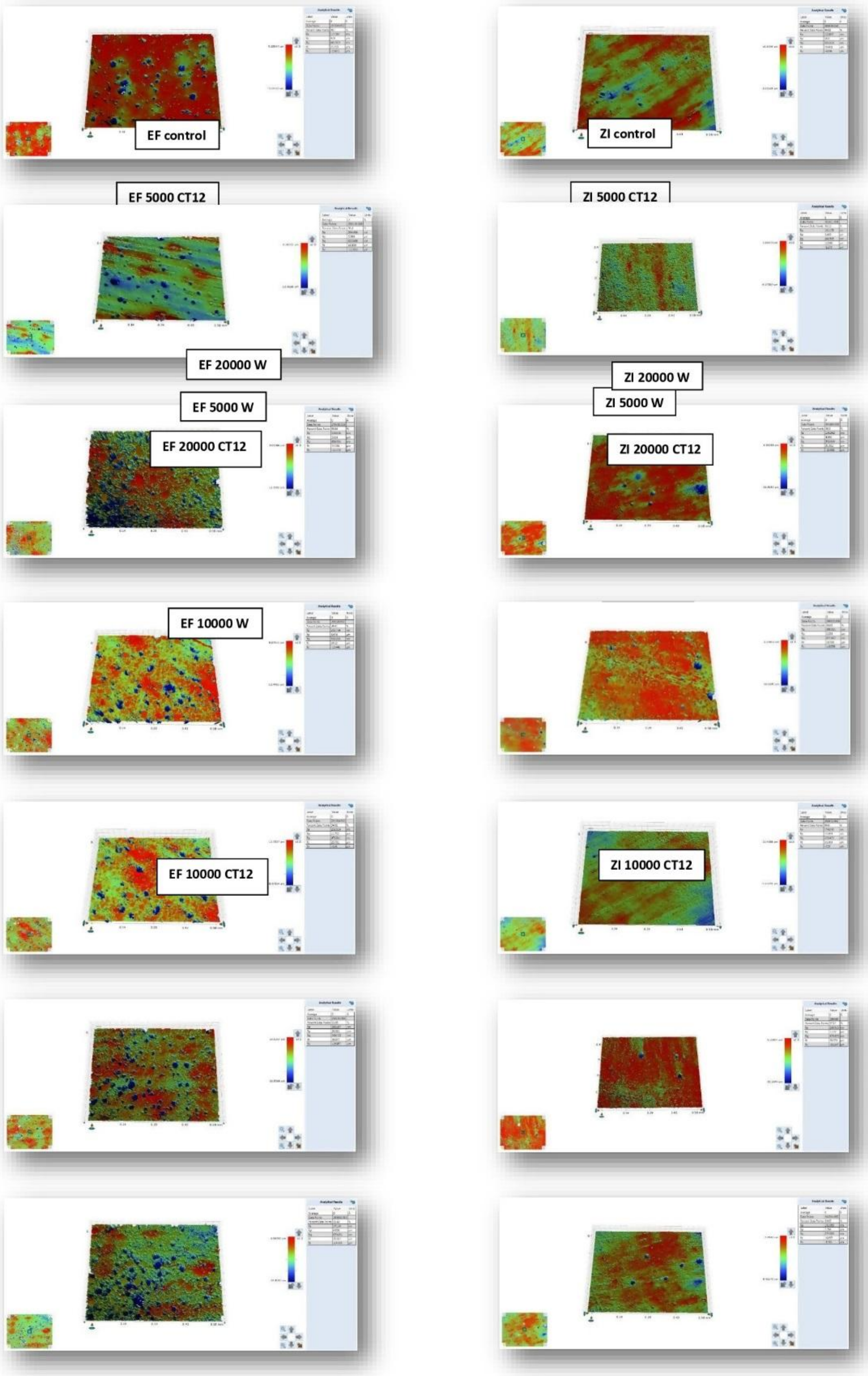

Fig-2: The figures above shows the profile of the surface roughness of the EF and ZI nano glass ionomers. The figures presents the condition of EF and ZI specimens in a control state and in a state after being subjected to the simulated tooth brushing procedure for about 5,000, 10,000 and 20,000 cycles using the only water wet tooth brush and using the wet tooth brush with and dentifrice (CT12), in detail analysis

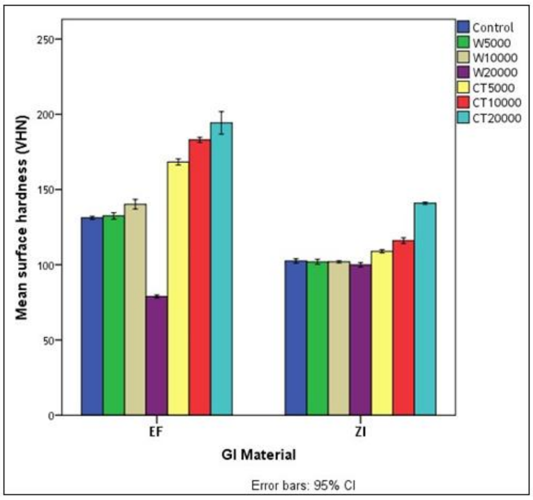

Fig-3: The figure represents the mean surface hardness (VHN) of the EF and ZI specimens under all the possible conditions

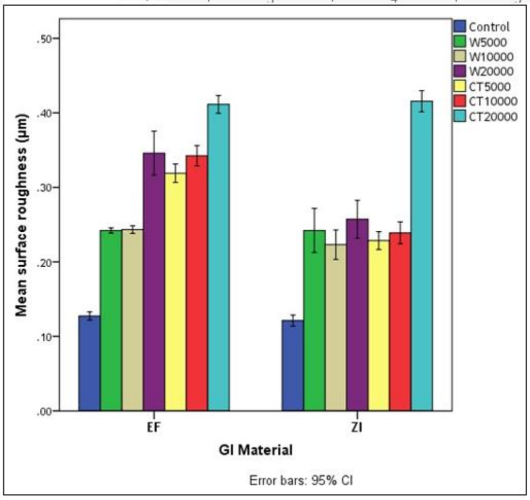

Fig-4: This above figure shows the mean surface roughness (µm) of the EF and ZI specimens under all the possible conditions

DISCUSSION

In the most recent years, the nanotechnology has expanded to a great extent in improving the composition of the restorative dental materials. It also improves the durability and the properties of the restorative dental materials. In this study, the Nano resin-coated GI Equia forte Fil (GC Corp., Tokyo, Japan) and Nano zirconia-reinforced GI Zirconomer Improved (Shofu Inc., Kyoto, Japan) were used. The current investigative study was conducted in order to make an attempt to measure and compare the tested GI materials regarding resistance to the surface roughness and surface hardness degradation after application of simulated tooth brushing for 5,000, 10,000 and 20,000 cycles, using water alone or water with a tooth dentifrice. All the possible factors that can affect the surface roughness and hardness were standardized at the time of application. The factors include; the type of dentifrice, the softness and number of the toothbrush bristles and the time and load applied in the simulated tooth brushing.

The Vickers Surface Hardness (VSH) was used in this research study which describes the surface resistance of a substance to permanent microscopic indentation under a loaded square pyramid-shaped diamond indenter. It is considered as an important and useful tool for testifying the brittle materials, such as GIs. The mutations in the composition e-g., particle shape and size of the material affects greatly.

The surface roughness of the substance can be defined in terms of the arithmetic average height of the surface irregularities from the mean line that must be evaluated with in the scanned length. The plague retention and the microbial colonization can be resulted when the dental restorations with the surface roughness exceeds the values of 0.2 µm. It may possibly lead to the plaque discoloration and secondary carries. At the level of 0.3µm surface roughness, the patient tongue discomfort including the dull and unsatisfactory appearance can be observed.

This research study revealed that the basic VHN is higher in Equia forte Fil than in the Zirconomer Improved. It may be possible due to the presence of the Nano resin-protective coat on the surface of the Equia forte Fil specimens. This Nano coat has a resinous component which is hard enough to resist the dissolving of GI matrix in the initial phases of the GI setting reaction. According to the statistical point of view, the changes in Equia forte Fil on its subjection to the simulated tooth brushing with water in about 10,000 cycles was not very significant. The Nano resin-protected coat present on the surface of the specimens is durable and efficient in their persistence. They help in setting the reaction to maturation after being sealed from the outside wet environment. Normally, the GIC has a sudden and quick setting properties but it matures with a passage of time.

Although, the outcomes of this study indicate that the mean VHN of the Equia forte Fil decreases significantly after subjected to the 20,000 cycles of the simulated tooth brushing with water only. It has been observed that this significant decrease can be the result of partial or complete loss of the hard protective Nano coat due to the large number of the brushing cycles, which in return exposes the soft GI matrix. The complex forces applied during extended tooth brushing might be transmitted to the inner matrix generally and to the fillers particularly since the fillers have a higher elastic modulus than the matrix. The transmission of these forces to the fillers might have resulted in the de-bonding of the fillers from the matrix and also created the higher concentration of the stresses. This might have caused the microcrack formation which in return resulted in the water absorption, surface softening and the solubility of the GI material.

On the other side of the reaction, when the Equia forte Fil was subjected to the tooth brushing procedure for 5,000, 10,000 and 20,000 brushing cycles with using the CT12 tooth dentifrice, it showed the significant increase in its mean VHN. This unexpected increase in the surface hardness could be attributed to the surface deposit precipitation from the CT12 on the surface of the Equia forte Fil specimens.

These obtained results agree with another recent research study that showed a significant increase in the surface hardness of the bovine tooth dentin after being subjected to 20,000 cycles of the same simulated tooth brushing procedure by using CT12. CT12 includes a well- known broad-spectrum antibacterial agent, triclosan in it. Triclosan is known as an uncharged molecule which has a tendency to rapidly disappear from the oral environment. Therefore, the polyvinyl methyl maleic acid (PVM/MA) copolymer is also added as an assisting molecule in this tooth dentifrice to act as a carrier for the triclosan. It aids in its retention in the oral cavity and increase its effectiveness.

The previous researches on the human teeth revealed that the PVM/MA could adhere to the enamel surfaces, it reduces the erosion, and occlude dentin tubules, in return forming the protection barrier that prevents the dentin erosion. It also contributes to the adhesion of dentifrice to the dentin surface by binding itself to the surface collagen through the weak chemical bonding. The results of another study showed that a dentifrice with almost the same chemical formula as CT12 had a significant anti-hypersensitivity effect on enamel and dentin. This dentifrice produced dentin surface deposits and tubule plugs containing silicon in addition to calcium and phosphorus, as indicated by energy-dispersive x-ray and electron spectroscopy for chemical analysis [14, 15]. The same concepts of surface deposit precipitation from CT12 on natural human enamel and dentin could apply to the EF GI material used in this study and was subjected to CT12 during simulated tooth brushing.

Regarding ZI, its mean VHN displayed insignificant reduction after simulated tooth brushing for up to 10,000 cycles using water, which is equivalent to 1 year of clinical use [16]. The ZI has a certain kind of resistance to the surface softening which could be attributed to the presence of nano-sized zirconia filler particles in its composition combined with the tiny inter-particle spacing present in its matrix. This composition particularly increased the surface resistance of the material plus the transformation toughening effect of the zirconia also added in increasing the strength of the material on its exposure to load. Thus, the improvement in overall strength properties of the zirconia acts as an assisting factor in increasing the resistance of the material to the surface degradation.

However, after subjected to simulated tooth brushing for up to 20,000 using water, ZI showed the significant reduction in its mean surface hardness. The ZI could not withstand the additional applied load during the procedure without affecting its surface hardness. This finding can be explained as: ZI is delivered in the form of powder and liquid as it is the water-based GIC. ZI showed the high viscosity level at the time of mixing which becomes responsible for the presence of the air bubbles and the pores in the matrix. Some of them constitute the passages to allow the water to penetrate the set of ZI matrix which caused the surface hydrolytic instability and softening. The load and the frictional shearing forces applied during tooth brushing were transmitted through the specimen surface to the resin matrix and then to fillers, as the fillers possess a higher modulus of elasticity than the resin matrix. The transmission of frictional forces to the fillers might have led to a weakening of the bond between the filler particles and the matrix.

At one end, this might have resulted in the dislodgement of some particles of the filler from surface, in return exposing the set matrix present under it. At the other end, the produced concentration of the stresses around the particles of the filler could possible lead to the microcrack formation. Both of the explained conditions eventually result in exposing through the pores and opening passages for water penetration, which in return caused the absorption in the ZI matrix set and the surface softening.

This research study revealed that the ZI showed a significant increase in the mean VHN when exposed to simulated tooth brushing by using CT12 as compared to its mean control. The reasons for the increase of VHN in ZI could be identical as for the EF in increasing its VHN. The concepts of surface deposit precipitation from the CT12 during the simulated tooth brushing procedure used for Equia forte Fil could be applicable to ZI specimens.

In case of the surface roughness, the results of the present research showed a very closely identical control mean Ra of the both Equia forte Fil and ZI, even after exposure to the 5,000 cycles of the simulated tooth brushing using a wet toothbrush. However, excessive increases or decreases in the surface hardness of EF and ZI after simulated brushing were mostly accompanied by an increase in surface roughness. These results are expected because of the assumption that the decrease in VHN is attributed to the surface effects caused by the friction between the surface of specimen and the bristles of the tooth brush.

This finding is expected, based on the assumption that reduction in VHN is due to surface effects caused by friction between the specimen surface and the bristles of the brush or the abrasiveness of the small, round, irregular particles of hydrated silica included in the composition of the CT12 dentifrice, which could lead to increased surface roughness [17]. Increases in VHN, which could be due to the precipitation of PVM/MA copolymer particles from CT12 in the form of surface deposits, might have an adverse effect on surface smoothness, leading to an increase in mean surface roughness. This explanation can also be applied to EF specimens subjected to 5,000 and 10,000 cycles of simulated tooth brushing using CT12.

However, EF specimens displayed higher mean Ra compared to the mean Ra of ZI specimens after 10,000 and 20,000 brushing cycles using CT12 or water alone. This higher mean Ra may be due to abrasive wear caused by friction of the brush bristles, leading to the plucking of filler particles from the surface. The greater the number of brushing cycles, the greater the surface roughness [18]. This proven finding explains the higher mean of Ra at 20,000 cycles than at 10,000. Moreover, the larger the size of lost filler particles during simulated brushing, the greater the surface roughness. The EF set matrix has micron-sized fluoro aluminosilicate filler particles compared to Nano-sized ceramic and zirconia filler particles in ZI. Thus, after simulated brushing, the lost filler particles from the surface of EF left larger spaces than those left due to lost counterparts from the surface of ZI. These larger spaces left on the surface of EF explain its rougher surface compared to ZI after extensive simulated brushing [19].

However, after 20,000 brushing cycles with CT12, the mean Ra of Equia forte Fil and Zirconomer Improved were significantly higher than their equivalents when the brushing was conducted with water only. These higher mean Ra may be due to the presence of deposits precipitated on specimen surfaces from the CT12 dentifrice as described above, which could also lead to an increase in surface roughness. These findings coincide with a recent study that found that the extended use of CT12 with simulated tooth brushing for 20,000 cycles led to a significant increase in the surface roughness of bovine tooth dentin [20].

CONCLUSION

Irrespective of the testing conditions, the EF showed the higher mean surface hardness and higher mean surface roughness as compared to the ZI. Both the EF and ZI specimens demonstrated the resistance to the softening when being subjected to the simulated tooth brushing procedure equivalent to about 1 year of the clinical use. Both the EF and ZI displayed the significant reduction in the surface hardness, after being subjected to the extensive simulated tooth brushing with water equivalent to two years of the clinical use. It has been showed that the use of the CT12 tooth dentifrice for tooth brushing that have EF and ZI restoration can cause an increase in their surface hardness. Both specimens used, EF and ZI, demonstrated the increases of concern regarding their surface roughness after being subjected to the simulated tooth brushing using water for a period equivalent to the 2 years of clinical use.

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