IN-VITRO WEAR AND HARDNESS OF NEW CONVENTIONAL GLASS IONOMER CEMENT COATED WITH NANO-FILLED RESIN

by

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DEDICATION

This thesis is dedicated to my mother, my brothers, and my sisters for their support and guidance since the beginning of my study.

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INTRODUCTION

Glass ionomer cement (GIC) was introduced to the dental field in the 1970s by Wilson and Kent. The cement was composed of ion-leachable fluoroaluminosilicate glass in a solution of polyacrylic acid that sets through an acid-base reaction. GIC combines the advantages of translucency and fluoride release from silicate cement, and the advantages of biocompatibility and desirable adhesive properties from polycarboxylate cement. These materials have several benefits when used as a direct restorative material. These include fluoride release and uptake; chemical bonding to tooth structure, and expression of similar coefficients of thermal expansion to the tooth structure. The main disadvantage of GIC is in its low-value mechanical properties when compared with other available restorative materials.

Efforts have been made to improve the material and increase its mechanical properties. Recently, GC America announced the launch of EQUIA, purported to be the first self-adhesive posterior restorative system. This system is composed of two major components, high-strength conventional GIC and a nano-filled, resin-based coating material said to provide a high gloss, smooth surface, and increased wear resistance for the restoration. The manufacturer claims this material can be used as a replacement of amalgam and composite resin when restoring class I, II and V cavities. The results of previous studies show that wear resistance of high-strength conventional GIC is inferior to composite resin.[1, 2] Evidence is needed to determine whether application of a nano-

filled surface resin will enhance the wear resistance and microhardness of high-strength GIC.

It was the primary objective of this *in-vitro* study to measure the wear resistance and hardness of EQUIA and compare it with other restorative materials.

REVIEW OF LITERATURE

HISTORICAL BACKGROUND

GICs were first introduced to the dental field in the 1970s by Wilson and Kent.[3] They tried to combine the advantages of translucency and fluoride release from silicate cement and the advantages of biocompatibility and adhesive properties from polycarboxylate cement. The result was cement composed of ion-leachable fluoroaluminosilicate glass in a solution of polyacrylic acid called GIC. The first commercially available GIC in the US was called ASPA (alumino-silicate poly-acrylate). It was only indicated to restore cervical caries or abrasion lesions due to low mechanical properties. In addition, it was hydrolytically unstable in the oral environment. Since that time, GICs have undergone considerable research development. Improvements in several physical and mechanical properties have been sought in an attempt to expand the applications of GIC in restorative dentistry.

BASIC CHEMISTRY

Conventional GICs are provided as a powder and a liquid.[4] The powder consists of fluoroaluminosilicate glass particles $SiO2$-AlO_3-CaF_2-$AlPO_4$-$NaAlF_{6,}$ which are fused at high temperature and then crushed and powdered to small-size particles. The liquid is an aqueous solution of mainly polyacrylic acid in addition to other acids added to improve the handling and setting of the cement. Tartaric acid is used in glass ionomer liquid to extend the working time and to strengthen the cement.[5] An acid-base reaction occurs when the powder and liquid are mixed together. As the liquid attacks the glass

particles, calcium and aluminum ions release, and metallic polyalkenoate salts start to form and begin to precipitate. Gelation occurs and then proceeds until the cement sets hard.[4]

Developments in GIC

In the late 1980s and early 1990s, resins were incorporated into the chemical composition of GIC to improve the handling characteristics and physical properties.[6] The resultant hybrid cement is called resin-modified GIC. In this type of cement, the powder is similar to that of conventional GIC. The main difference is in the liquid composition. Polymerizable methacrylate groups are added to the polyacrylic acid to supplement the fundamental acid-base reaction by a second resin polymerization initiated either chemically or by light curing. An early commercial resin-modified GIC (Vitrebond) was introduced into the market by 3M in 1989 to be used as a liner or base under restorative materials.

Some differences in mechanical and physical properties were found between the conventional GICs and the resin-modified GICs. These differences were inconsistent and were different from product to product. DeGee and colleagues compared the wear resistance of conventional and resin-modified GICs. They found that resin-modified glass ionomers wore significantly faster than the conventional glass ionomers.[7] They also differed in fluoride release capability. Conventional GICs were found to release more fluoride than resin modified cements.[8]

Another method used to improve the mechanical and physical properties of GIC without affecting the fluoride release was by changing the chemical composition. Several consecutive studies were done by Crisp and colleagues in this field.[9-11] In the first study,

the effect of the powder/liquid ratio on the physical properties was evaluated. It was concluded that the highest powder/liquid ratio should be used that will allow for convenient mixing and handling.[7] In another study, the effect of polyacid concentration on improving the physical properties was investigated. The results suggest that it is desirable to use a high polyacid concentration.[8] A follow-up study on the effect of polyacid molecular weight on physical properties found that increasing the molecular weight of the polyacid resulted in a stronger cement with enhanced mechanical properties while increasing the viscosity of the cement, limiting the effectiveness of this strategy.[9] The results of these studies contributed to the introduction of new high-strength glass ionomer materials. These included products such as Fuji IX GP and Ketac-Molar. Studies on these materials have shown they improve mechanical and physical properties and clinical performance in comparison with conventional GICs, but that they lack even more in the esthetic appearance.[12-16]

Advantages of GIC as a Restorative Material

GICs have several advantages when used as a restorative material. These advantages include fluoride release and uptake, thermal expansion similar to that of tooth structure, adhesion to tooth structure, and biocompatibility.

Fluoride release and uptake is one of the most important advantages for GICs. Unlike calcium and aluminum, fluoride is not an integral part of glass ionomer matrix formation. Located mainly in the glass, the fluoride is available for release even after complete setting of the cement without affecting the structure of the cement. In addition, GIC may act as a reservoir for fluoride ions by taking up the ions after different

applications of topical fluoride and then making those ions available for release over a relatively long period of time.[17, 18]

Fluoride release makes the tooth area around glass ionomer restorations less susceptible to secondary caries. Several *in-vitro* and *in-vivo* studies examined the effect of fluoride release on enamel demineralization and caries experience.[19-22] Ostrom and colleagues in their *in-situ* study concluded that fluoride increases the enamel resistance to demineralization by acid attack. [20] Kotsanos in his *in-situ* study paired bovine enamel slabs with four different restorative materials and inserted them in flanges of dentures, inside the mouth, for 70 days. Results of this study showed that fluoride in a glass ionomer restorative material can inhibit caries formation in adjacent enamel surfaces. [19] In another *in-situ* study, Yamamoto and colleagues concluded that the presence of fluoride-containing GIC can enhance the remineralization in the immediate adjacent enamel surface. [22] Tyas in his *in-vivo* study assessed the cariostatic effect of GIC by comparing glass ionomer with composite resin in class V cervical cavities for five years. [21] The results showed that glass ionomer restorations have less chance to develop secondary caries when compared with resin composite. Another study done by Ten Cate and Van Duinen showed hypermineralization of dentin lesions adjacent to glass ionomer restorations, while specimens with amalgam or composite restorations showed further extensive demineralization.[23]

One of the advantages of GIC as a restorative material is the ability of the cement to adhere chemically to dental tissue by an ionic exchange at the interface. The polymer chains of the cement enter the molecular surface of hydroxyapatite and form metal ion bridges between calcium and phosphate ions.[24] It was shown that GIC bonds chemically

to enamel more than dentin because of a higher mineral concentration in enamel than in dentin.[25] This chemical adhesion helps minimize the need to create retentive form during cavity preparation and makes GIC suitable for use in atraumatic restorative treatment, which is based on removing decalcified tooth tissue and restoring the cavity with an adhesive filling material.

The oral cavity is subjected to a wide range of temperature changes when eating and drinking hot and cold foods. This causes the tooth structure and the restorative materials to expand and contract as the temperature changes. Mismatch in thermal expansion and contraction between the tooth structure and the restorative material lead to several problems, including microleakage and debonding in some cases.[26] One of the advantages of GICs is that their coefficient of thermal expansion is similar to that of tooth structure. Yan and colleagues in their study compared the coefficient of thermal expansion of different restorative materials.[27] They concluded that dimensional changes of conventional GICs are minimal compared with resin-modified GIC and composite resin.

GIC is considered a biocompatible material that can be safely used in deep cavities. Several studies have been done to assess the biocompatibility of GIC.[28-33] Results from these studies show that conventional GIC has very minimal effect on the pulp tissue. Furthermore, applications for GIC have extended into the medical field where it has been used as bone cement due to its high biocompatibility.[28]

In addition to the previously mentioned advantages, GIC cement is a tooth-colored material suitable for restorations when esthetics is a concern. Although not as

esthetic as composite resin, GIC is still considered more esthetic than metallic-colored materials.

Disadvantages of GIC as a Restorative Material

Major disadvantages of GIC in use as a restorative material include its weak mechanical properties, such as low wear resistance and low fracture toughness.[34] This makes it unsuitable for use in high-stress areas such as Class I and II restorations.

WEAR IN DENTISTRY

Wear is a common phenomenon that occurs when two or more surfaces undergo slipping or sliding movements as a load is applied. As surfaces slide over each other under different conditions, different mechanisms of wear can occur. Mair explained six mechanisms of wear.[35] These are abrasive, adhesive, fatigue, erosive, corrosive and fretting wear. Abrasive wear occurs when a hard surface slides against a softer surface and results in loss of structure of the softer surface. Adhesive wear occurs when asperities on one surface become cold-welded to the other surface as a result of friction and results in movement of material from one surface to another. Fatigue wear occurs as a result of subsurface crack propagation under dynamic load. Erosive wear happens when a material loses particles in an erosive medium under pressure. Corrosive wear is defined as loss of corrosion products from a material as it rubs against an opposing surface. Finally, fretting wear occurs when surfaces slip against each other slowly and for long periods of time under a load. In the oral cavity, wear can result from direct contact between the teeth (two- body wear) and from any abrasive particle or device between them (three-body wear) during mastication or parafunctional movements. The wear that occurs in the oral

cavity is complex and can involve different types of the previously mentioned wear mechanisms.

Wear resistance is an important property for all restorative dental materials. It shows the ability of the material to withstand grinding and the force applied to it from an opposing tooth, while maintaining its restoration form and function. Researchers have become increasingly interested in the simulation of wear mechanisms that occur in the oral cavity in an attempt to develop a wear-testing device to evaluate and compare the wear resistance of different dental materials. Several wear simulation and testing machines have been used in this field. One of the commonly used wear simulator machines is the toothbrush abrasion machine. It simply simulates the wear mechanism that occurs during tooth brushing. Another frequently used machine is the Alabama wear simulator. It simulates the three-body wear mechanism that occurs between opposing teeth in the presence of an abrasive medium. Several studies have been done to evaluate and compare the wear resistance of different restorative materials using either the toothbrush abrasion machine or the Alabama wear simulator, or both. [36-40]

Wear Resistance of GICs

Several studies and reviews have been done to measure the wear resistance of GICs and compare them to other restorative materials.[41-45] Sulong and Aziz reviewed the literature and concluded that GICs had much less wear resistance compared with other restorative materials.[44] In another study by Xie and colleagues, wear resistance of several commercially available GICs was evaluated and compared with composite resin.[45] They found that the most wear-resistant glass ionomer was still much lower in wear resistance than composite resin. Momoi and colleagues compared the wear of different GICs with

amalgam and composite resin.[46] Results of their study showed that both conventional and resin-modified GICs had significantly much higher wear than composite resin and amalgam. Results also showed that resin-modified GICs had higher wear than the conventional GICs. Forss and colleagues compared the abrasion resistance and surface hardness of four GICs.[47] They used composite material, enamel, and dentin as controls. They found that all glass ionomers investigated showed greater wear than composite or enamel, but less wear than dentin. They also found that all GICs investigated had lower hardness values than the composite. Smales and Joyce compared the abrasion resistance of GIC to composite resin and found that GIC abraded three times as rapidly by volume as composite when tested by a two-body abrasion method.[48] Schmage and colleagues evaluated the wear and hardness of different restorative materials, including GICs and resin composite.[49] They used two-consistency GICs, regular and packable. Results show that the wear of both GICs was significantly higher than that of restorative composites. They also found that packable GIC had higher hardness than the regular cement but that both were significantly lower than dentin.

MICROHARDNESS

Microhardness is an important physical property of a dental material.[50] It is defined as the resistance of a material to indentation or penetration. It is indicative of the ease of finishing of the material and its resistance to scratching.[51] The microhardness of glass ionomer has been evaluated in several studies for different purposes. This property has been used to evaluate the setting behavior and depth of cure of resin-modified GICs.[52] Others compare the hardness of GIC cement with other restorative materials and relate the hardness numbers to the microstructure of the material.[45] Gladys and colleagues

reviewed the literature about resin-modified GICs and concluded that resin-modified GICs were not indicated for occlusal restorations in the dentition, because the cements' surface hardness is too low compared with that of enamel.[53] Schmage and colleagues found in their study that packable GIC had higher hardness values than the conventional cement, but both were significantly lower than values for resin composite and dentin.[49]

IMPROVEMENTS IN GICs

GICs exhibit several unique properties as mentioned above. However, their low wear resistance makes them unsuitable to be used in high stress areas. Efforts have been made to improve the material and increase their mechanical properties. Several methods have been used in an attempt to improve the properties. A recent method includes coating the surface of the GIC with different coating materials as suggested in a recent product release by GC America, Inc.

In November 2009, GC America announced the launch of EQUIA, purporting it to be the first self-adhesive posterior restorative system. This system is composed of two major components, Fuji IX GP Extra and G-Coat Plus. The first component (Fuji IX GP Extra) is a packable self-cure conventional GIC with glass fillers that provide durable translucency and esthetics. The second component (G-Coat Plus) is a nano-filled, resin-based coating material said to provide a high-gloss, smooth surface and increased wear resistance for the restoration. The manufacturer claims this material can be used as a replacement for amalgam and composite resin when restoring Class I, II and V cavities.

Kato and colleagues evaluated the influence of various coating materials on flexural strength of conventional GIC.[54] They compared uncoated cement, cement coated with unfilled resin, and cement coated with nano-filled resin. Results of their study

showed that the nano-filled resin coat had the highest bond strength to the surface of GIC among the examined coating materials. Results also showed that the resin coat improved flexural strength of substrate restorative GIC.

Magni and colleagues studied another aspect of coating the GIC restoration with resin coat.[55] They utilized a SEM observation and a microleakage test to evaluate the marginal integrity of class V restorations. They found no gap in coated restorations and recommended the coating procedure in class V restorations to reduce gingival microleakage.

RATIONALE FOR THIS STUDY

Evidence is lacking as to whether application of a nano-filled surface resin will enhance the wear resistance and microhardness of high-strength GIC. The primary objective of this *in vitro* study was to measure the wear resistance and hardness of a high-strength glass ionomer coated with a nano-filled surface sealant and to compare it with other restorative materials.

MATERIALS AND METHODS

STUDY DESIGN

The wear resistance and hardness of four different restorative materials were measured and compared in this *in-vitro* study. Brand names, batch numbers, manufacturers, and composition of the tested products are listed in Table I. The resin composite Z-100 and a conventional GIC Fuji IX GP Extra were used as controls.

Three different laboratory tests were conducted on the four materials:

1. Toothbrush abrasion test.
2. Three-body Alabama wear test.
3. Knoop microhardeness test.

After testing, the worn surface of the specimens was observed with a metallograph to visually analyze the wear surfaces involved. In addition, selected EQUIA specimens were sectioned and examined with a metallograph to evaluate the thickness of the coating.

TOOTHBRUSH ABRASION TEST

Toothbrush abrasion was conducted in a method similar to that used by Jain and colleagues.[56] Six specimens of each material were prepared and tested. A custom-made stainless steel mold was used to fabricate six specimens of each material with the following dimensions: 2 mm, 5 mm and 25 mm. The materials were placed in the mold in one increment and covered with a Mylar strip. They were pressed with a cover glass slide to ensure the material was flush with the surface of the mold. For the Z-100 and Fuji II LC groups, the specimens were light-cured with a LE Demetron II curing light (Kerr

Corp., Orange, CA) having a light output of 820 mW/cm^2 in three different locations, each for a time of 40 seconds for Z-100 and 20 seconds for Fuji II LC to ensure covering of the entire specimen before removal of the strip. For the EQUIA and Fuji IX GP Extra groups, the materials were allowed to set for 10 minutes before removal of the strip. For the EQUIA group, after removal of the strip, the surface of the specimen was coated with G-coat using a small brush and then cured for 20 seconds each time in three different locations to ensure covering of the entire specimen. The side surfaces of the specimens were then finished with silicon carbide paper through 800-grit. After finishing, the specimens were stored in distilled water at 23^{o}C for 24 hours. Specimens were kept in a 100-percent humidity environment and weighed to an accuracy of ±0.1 mg until a constant mass (M_1) was obtained. The volume of each specimen was calculated by measuring and multiplying its dimensions using a Mitutoyo Digimatic Caliper (Mitutoyo America Corp., Aurora, IL) to an accuracy of 0.01 mm (V_1). The density (d) of each specimen was then calculated according to the following equation:

$$d = M_1 / V_1$$

The specimens were brushed in a mechanical tooth brushing machine (Pepsodent Co., Chicago, IL) (Figure 1) with a frequency of 170 strokes per minute for two hours (total of 20,400 strokes) under a 2.3-N vertical load. Aqueous slurry of Colgate Total tooth paste and water with a proportion of 1:1 by weight was used during the brushing. After completion of the brushing cycles, specimens were removed, cleaned with distilled water, and kept in a 100-percent humidity environment and weighed every 24 hours until a constant mass (M_2) was obtained. The new volume (V_2) and the amount of volume loss (ΔV) were then calculated using the following equations:

$$V_2 = M_2 / d$$

$$\text{Volume loss } (\Delta V) = V_1 - V_2$$

Three-Body Alabama Wear Test

The test was conducted in a method similar to that used by Jain and colleagues.[56] Eight cylindrical specimens of each material with a 9.5-mm diameter and a 3-mm thickness were prepared and tested using the Alabama wear machine (Figure 2). The materials were placed in the specimen holder in one increment except for the composite resin, which was placed in 2-mm increments. The materials were slightly overfilled. A piece of a Mylar strip was placed on top of the material and covered with a glass slide and pressed down to make a flat surface. For the Z-100 and Fuji II LC groups, the specimens were light-cured with an LE Demetron II curing light (Kerr Corp., Orange, CA) for 40 seconds for Z-100 and 20 seconds for Fuji II LC in one exposure before removal of the strip. For the EQUIA and Fuji IX GP Extra groups, the materials were allowed to set for 10 minutes before removing the strip. Specimens were then finished with SiC discs on a polishing wheel in the order of 400-grit, 600-grit and 800-grit.

For the EQUIA group, after finishing the specimens, the surface of each specimen was coated with G-coat using a small brush and then recovered with the strip and cured for 20 seconds to ensure a smooth flat surface. The specimens then were stored in distilled water at 23^{o}C for 24 hours.

A digital micrometer (Nikon Digimicro ME05, New York, NY) was used to measure the slider height on the machine before the test and then screwed into the pistons. The specimen holders were placed into an acrylic specimen holder chamber and secured into place to prevent any movement of the specimen (Figure 2). A testing

medium was prepared by mixing 15.0 grams of orthodontic resin powder (Dentsply Caulk [Lot #070924]) with 9 ml of distilled water that was then placed over the specimens. The load on the piston was adjusted to 75 N of pressure and the speed set to 75 revolutions per minute. The machine was set to perform 400,000 cycles.

The specimens were removed and cleaned with distilled water. On each specimen, a dot was put close to the top edge of the holder and again at positions of 45°, 90°, and 135°. A small clear ruler was used to draw four straight pencil lines across the surface of the specimen and the holder connecting the four dots. The intersection of the lines was considered the center of the worn area, and with the use of a Surtronic 3+ profilometer, the lines guided four surface profiles for each specimen (Surtronic 3+, Taylor Hobson Pneumo, Leicester, England). Area curves were obtained for each profile with the aid of TalyProfile Lite software version 3.1 (Taylor-Hobson). The software calculated the area under the curve and the amount of volume loss in millimeters cubed.

MICROHARDNESS TEST

The test was conducted in a method similar to that used by Roberts and colleagues.[52] A cylindrical brass mold with dimensions of 6 mm diameter and 1 mm thickness was used to fabricate five specimens of each material. The mold was placed on a glass slide covered by a Mylar strip and filled with the material. It was covered with another Mylar strip and pressed with a cover slide to ensure the material was flush with the surface of the mold. For the Z-100 and Fuji II LC groups, the specimens were light cured using a LE Demetron II curing light (Kerr Corporation, Orange, CA) for 40 seconds for Z-100 and 20 seconds for Fuji II LC, before removing the strip. For the EQUIA and Fuji IX GP Extra groups, the materials were allowed to set for 10 minutes

before removing the strip. For the EQUIA group, after removal of the strip, the surface of the specimen was coated with G-coat using a small brush and then cured for 20 seconds. After that, the specimens were stored in distilled water at 37^{o}C for 24 hours.

The hardness of the specimens was measured using a microhardness testing machine (M 400, Leco, St. Joseph, MI) (Figure 3). The Knoop hardness test was performed using a diamond indenter with a 100-g load and 10-sec dwell time. Microhardness measurements were made on three randomly selected spots on the top surface of the specimen and the mean of the three values was calculated and used as the surface hardness of the specimen.

SPECIMEN PREPARATION FOR METALLOGRAPH EXAMINATION

After completing all tests, selected specimens were chosen from the Alabama test and three from the toothbrush abrasion test to be examined under the metallograph (Leco Metallograph, St. Joseph, MI).

In the beginning, the surface of each specimen was directly examined under the metallograph and a micrograph was taken with a digital camera (Digital Microscope Camera DMC 1, Polaroid, Cambridge, MA). Specimens were then embedded in self-cure acrylic and sectioned in the middle of the specimen with a water-cooled precision saw (Isomet 1000 Precision Saw, Buehler, Lake Bluff, IL). Cross-sectional specimens were then cleaned in an ultrasonic cleaner for 15 minutes and examined under the metallograph to evaluate the thickness of the coating material on worn and unworn areas.

STATISTICAL METHODS

For each test, summary statistics (mean, standard deviation, standard error, range) were calculated for each of the four materials. The materials were compared for differences in Alabama wear test volume loss, toothbrush abrasion volume loss, and micro hardness (KHN) using one-way analysis of variance (ANOVA). Pair-wise comparisons among the groups were made using Tukey's method to control the overall significance level at 5 percent. Given the Alabama-wear-test volume loss was not normally distributed, the comparisons among materials were performed using Wilcoxon Rank Sum tests. For these tests, Sidak's method was used to control the overall significance level of the pair-wise comparisons at 5 percent.

RESULTS

TOOTHBRUSH ABRASION RESULTS

Table II and Figure 4 show the means and standard deviations of volume loss resulting from the toothbrush abrasion test. The mean average volume loss values for Z-100, EQUIA, Fuji IX GP Extra and Fuji II LC were 3.198 mm^3, 3.129 mm^3, and 3.283 mm^3 respectively. Although there are some differences among these materials, these differences were not significant. Fuji II LC showed the statistically highest amount of volume loss with an average volume loss of 4.959 mm^3.

THREE-BODY ALABAMA WEAR RESULTS

The means and standard deviations of volume loss resulting from the three-body Alabama wear test are summarized in Table III and Figure 5. The mean average volume loss for EQUIA and Z-100 was 0.289 mm^3 and 0.137 mm^3 respectively. The difference was not significant between Z-100 and EQUIA. Fuji IX GP Extra had significantly more volume loss than both EQUIA and Z-100. Fuji II LC showed an average volume loss of 2.627 mm^3, which was statistically the highest amount of volume loss among the materials tested.

KNOOP MICROHARDNESS RESULTS

The means and standard deviations of the Knoop Hardness Number (KHN) for the four materials tested are shown in Table IV and Figure 6. The mean average KHN for Z-100 was 82.33, which was significantly higher than for EQUIA, Fuji IX GP Extra and

Fuji II LC. Fuji IX had a significantly higher KHN than Fuji II LC and EQUIA, while Fuji II LC and EQUIA were not significantly different from each other.

TABLES AND FIGURES

TABLE I

Materials used in the study

Material type	**Material brand name**	**Batch #**	**Manufacturer**
Resin composite	Z-100	5904A2	3M ESPE Dental
High strength conventional glass ionomer cement	Fuji IX GP Extra	002535	GC America Inc.
Resin-modified glass ionomer cement	Fuji II LC	000139	GC America Inc.
Resin-coated glass ionomer cement	EQUIA	439402	GC America Inc.

TABLE II

Mean and standard deviation of material loss
obtained from toothbrush abrasion test

Material	**Average loss (mm^3)**	**SD**
Z100	3.198 [a*]	0.978
EQUIA	3.129 [a*]	0.637
Fuji IX	3.283 [a*]	0.709
Fuji II LC	4.595 [b*]	0.911

***** Values with similar letters are not statistically different.

TABLE III

Mean and standard deviation of material loss obtained from three-body Alabama wear test

Material	**Average loss (mm^3)**	**SD**
Z100	0.137 [a*]	0.045
EQUIA	0.289 [a*]	0.333
Fuji IX	1.061 [b*]	0.709
Fuji II LC	2.627 [c*]	0.911

* Values with similar letters are not statistically different.

TABLE IV

Mean and standard deviation of KHN obtained from microhardness test

Material	Average KHN	SD
Z100	82.333 [a*]	1.440
EQUIA	24.240 [c*]	2.859
Fuji IX	40.340 [b*]	4.436
Fuji II LC	29.213 [c*]	3.208

* Values with similar letters are not statistically different.

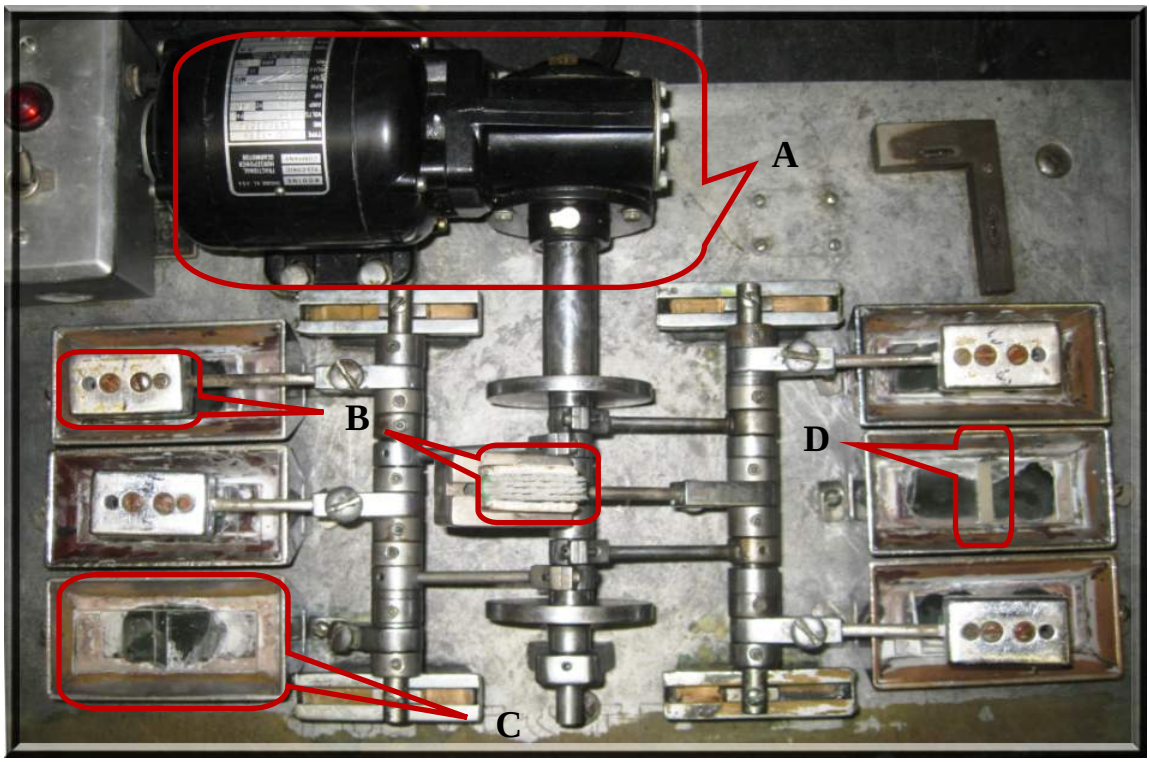

FIGURE 1. Toothbrush abrasion machine. A) Motor drive. B) Brush head. C) Specimen holder. D) Specimen.

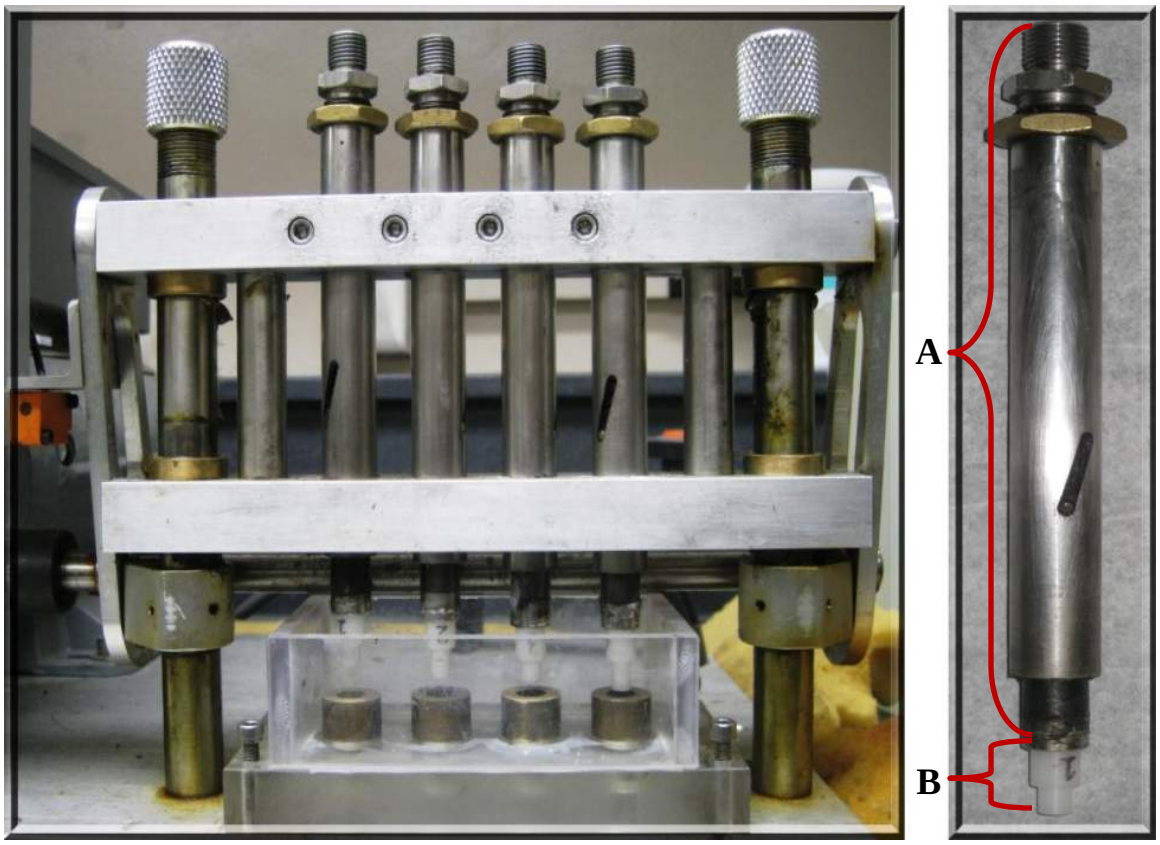

FIGURE 2. Three-body Alabama wear machine. A) Piston. B) Polydactyl slider.

FIGURE 3. Microhardness tester.

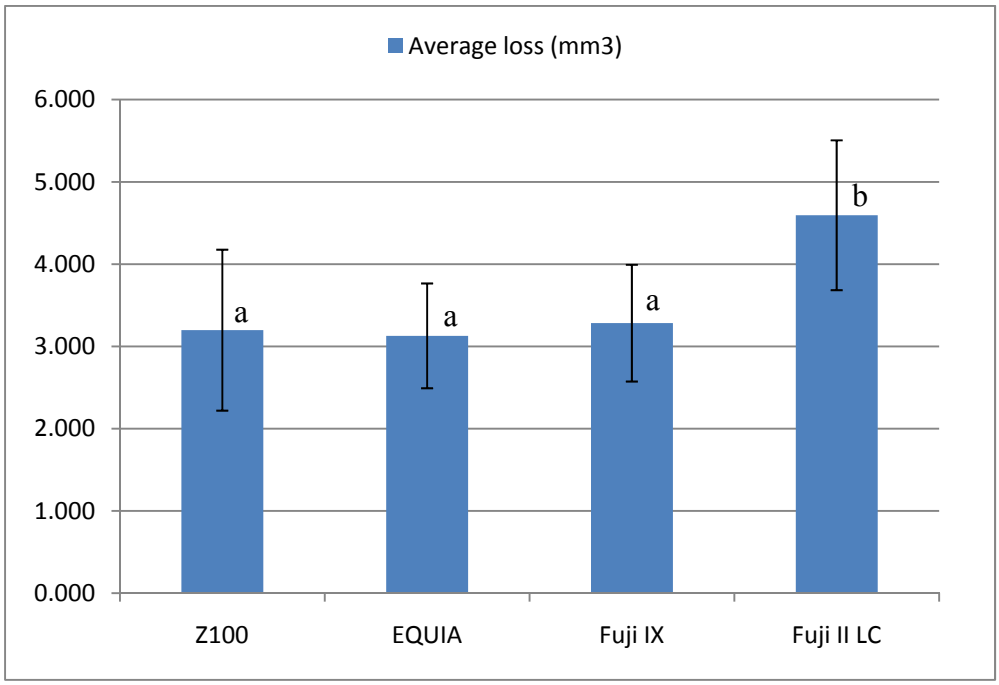

FIGURE 4. Mean volume loss and standard deviation obtained from toothbrush abrasion test. *Materials with similar letters are not statistically different.

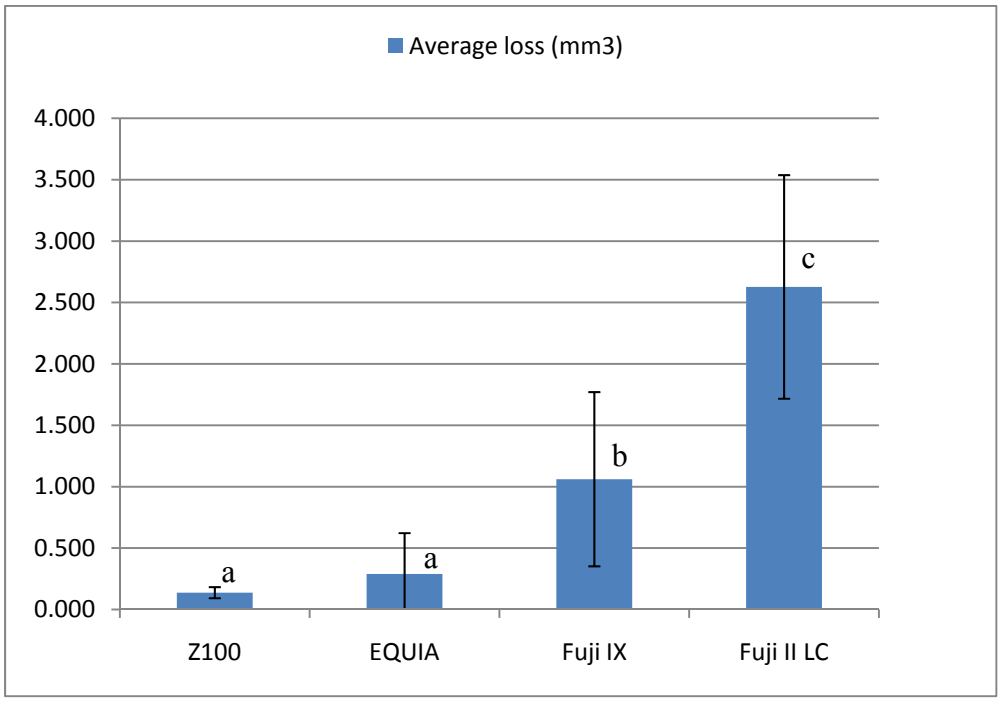

FIGURE 5. Mean volume loss and standard deviation obtained from three-body Alabama wear test. *Materials with similar letters are not statistically different.

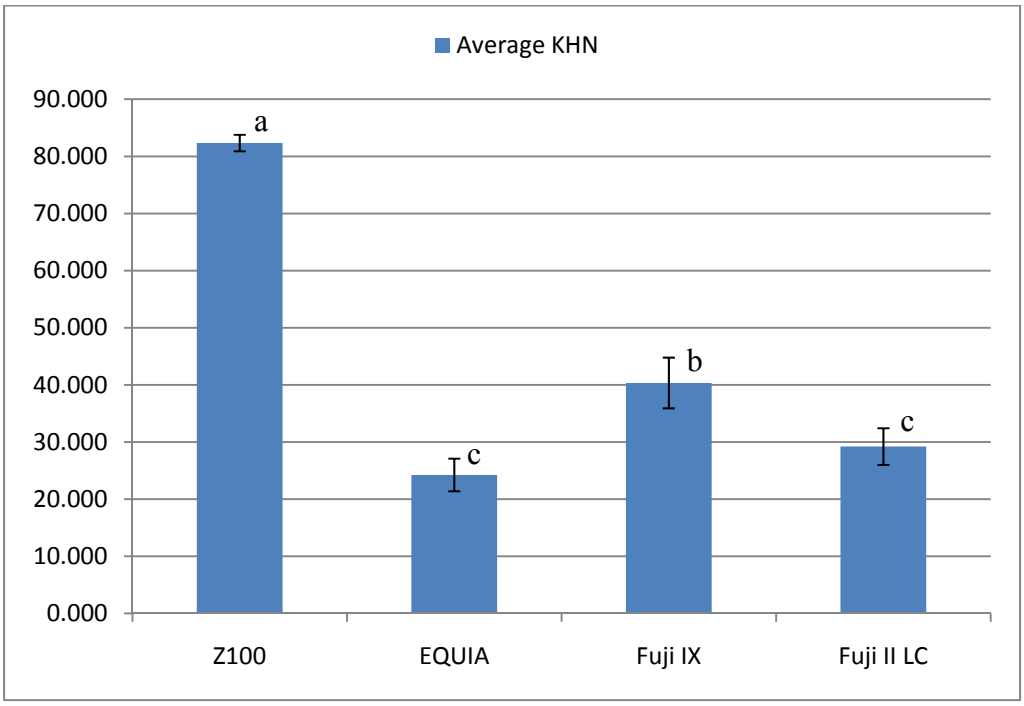

FIGURE 6. Mean KHN values and standard deviation obtained from microhardness test. *Materials with similar letters are not statistically different.

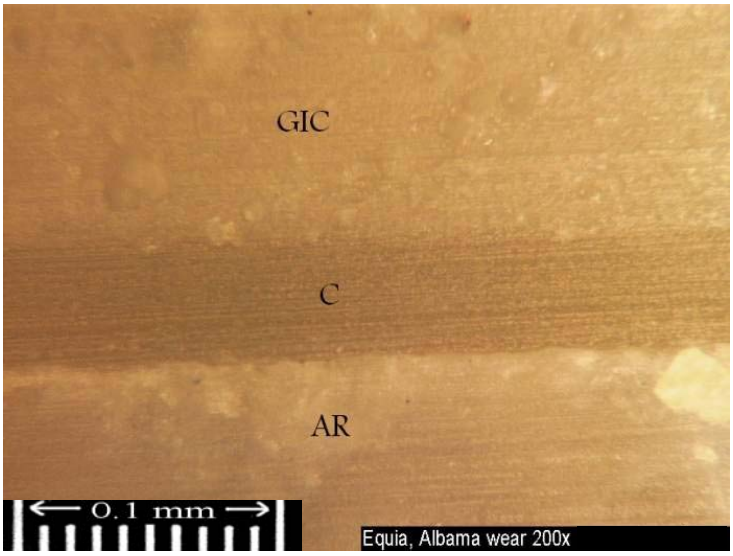

FIGURE 7. Cross-sectional micrograph of EQUIA specimen before three-body Alabama wear test. (Specimen 1) (GIC= glass ionomer; AR= acrylic resin; C= nano-filled resin coat)

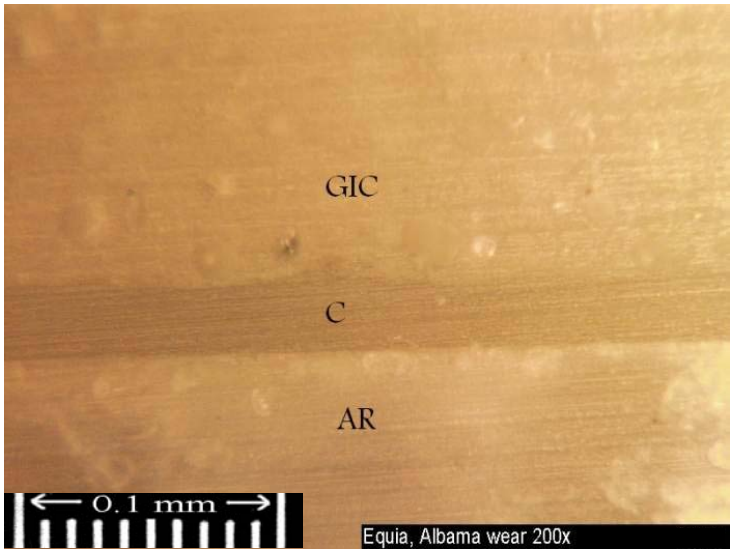

FIGURE 8. Cross-sectional micrograph of EQUIA specimen after three-body Alabama wear test. (Specimen 1) (GIC= glass ionomer; AR= acrylic resin; C= nano-filled resin coat)

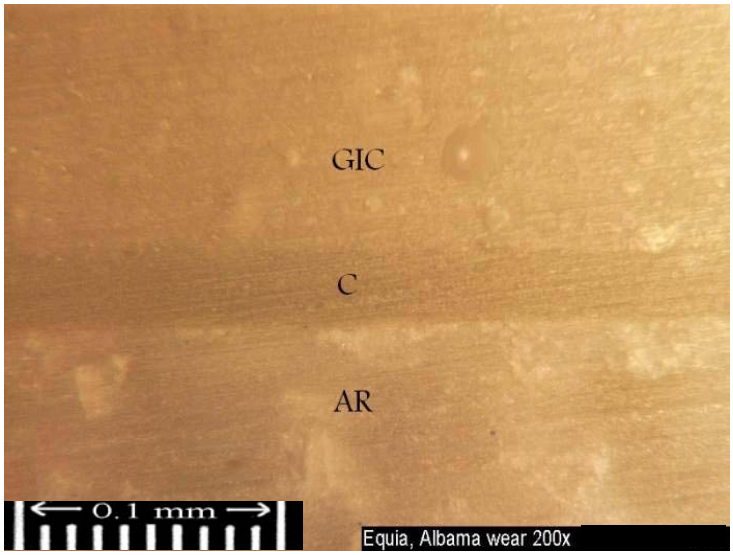

FIGURE 9. Cross-sectional micrograph of EQUIA specimen before three-body Alabama wear test. (Specimen 2) (GIC= glass ionomer; AR= acrylic resin; C= nano-filled resin coat)

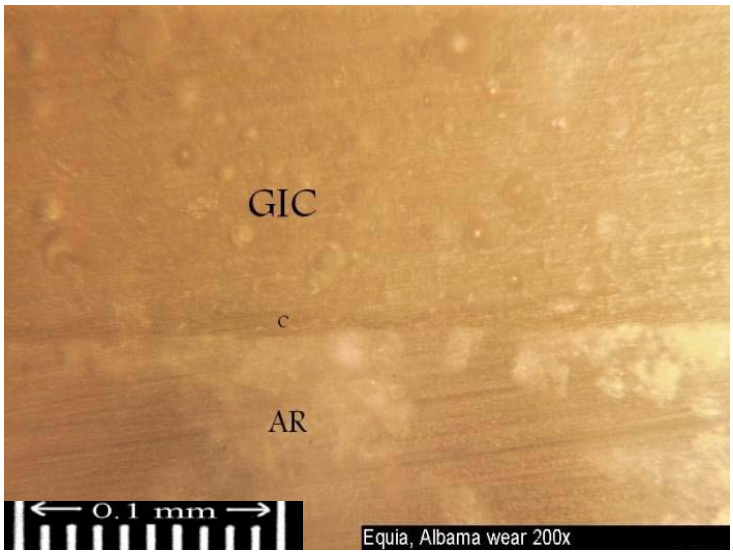

FIGURE 10. Cross-sectional micrograph of EQUIA specimen after three-body Alabama wear test. (Specimen 2) (GIC= glass ionomer; AR= acrylic resin; C= nano-filled resin coat)

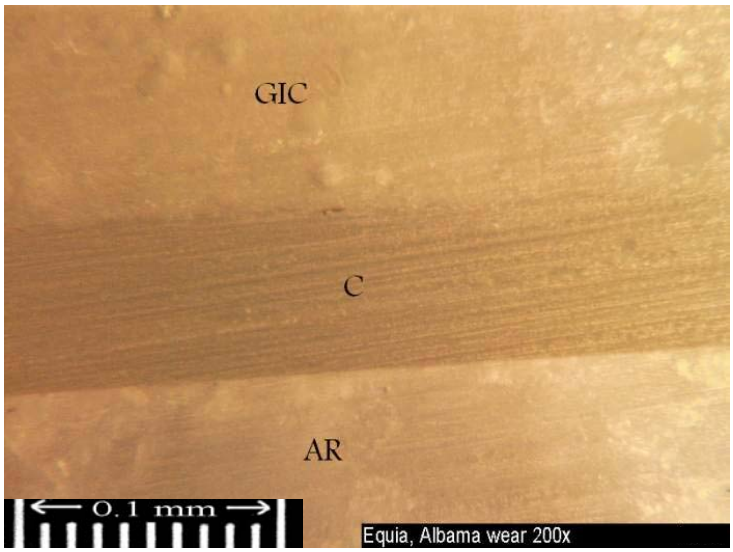

FIGURE 11. Cross-sectional micrograph of EQUIA specimen before three-body Alabama wear test. (Specimen 3) (GIC= glass ionomer; AR= acrylic resin; C= nano-filled resin coat)

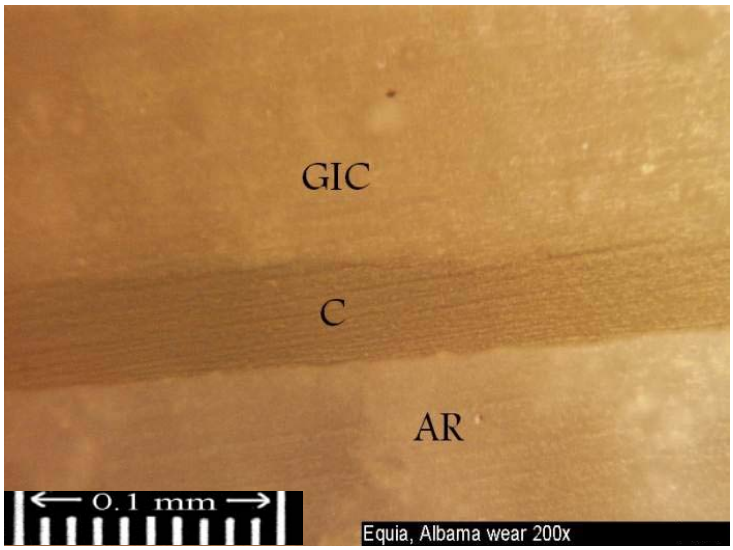

FIGURE 12. Cross-sectional micrograph of EQUIA specimen after three-body Alabama wear test. (Specimen 3) (GIC= glass ionomer; AR= acrylic resin; C = nano-filled resin coat)

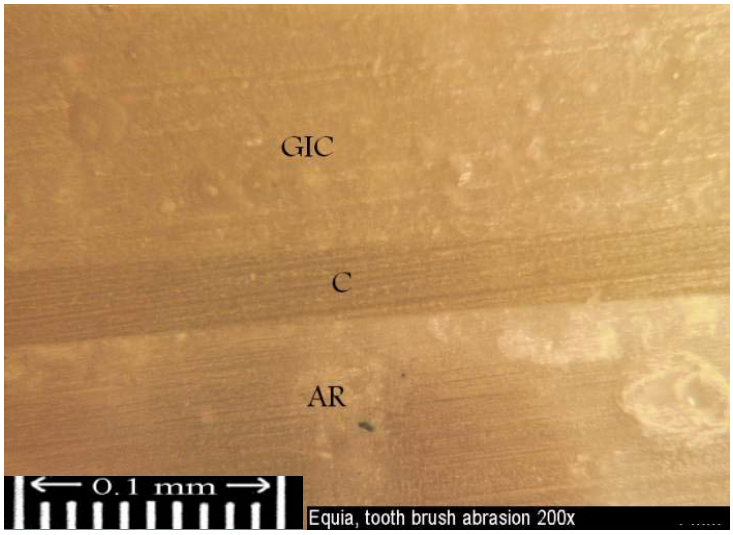

FIGURE 13. Cross-sectional micrograph of EQUIA specimen before toothbrush abrasion test. (Specimen 1) (GIC= glass ionomer; AR= acrylic resin; C= nano-filled resin coat)

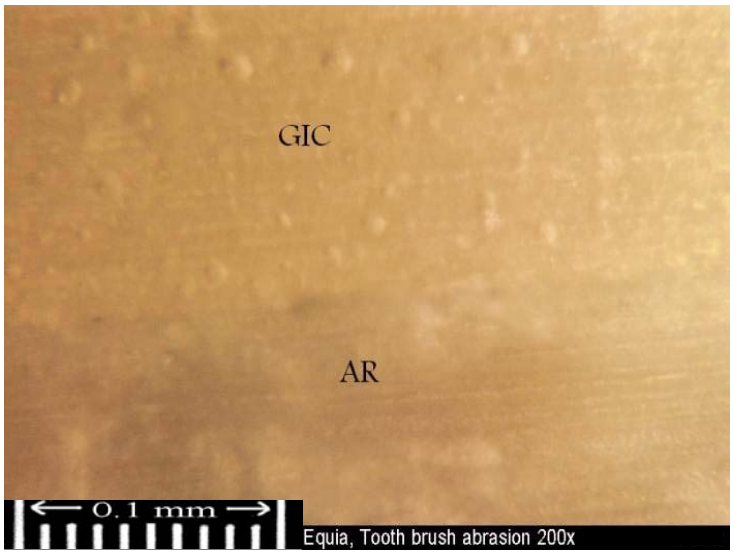

FIGURE 14. Cross-sectional micrograph of EQUIA specimen after toothbrush abrasion test. (Specimen 1) (GIC= glass ionomer; AR= acrylic resin)

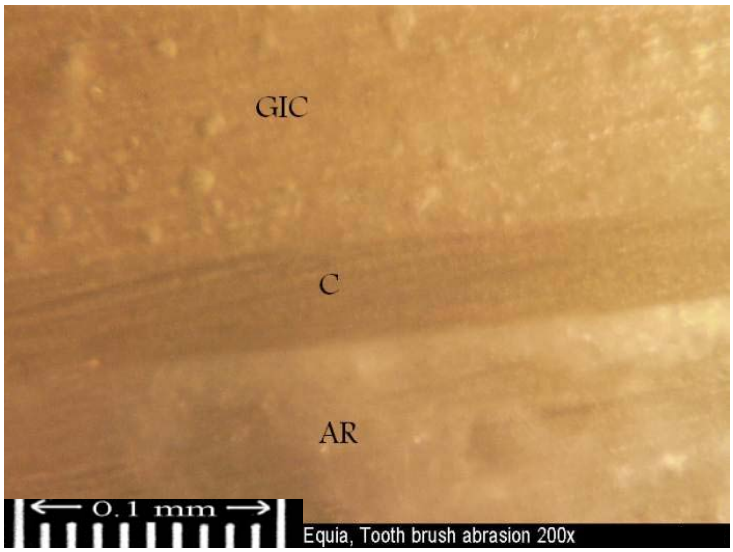

FIGURE 15. Cross-sectional micrograph of EQUIA specimen before toothbrush abrasion test. (Specimen 2) (GIC= glass ionomer; AR= acrylic resin; C= nano-filled resin coat)

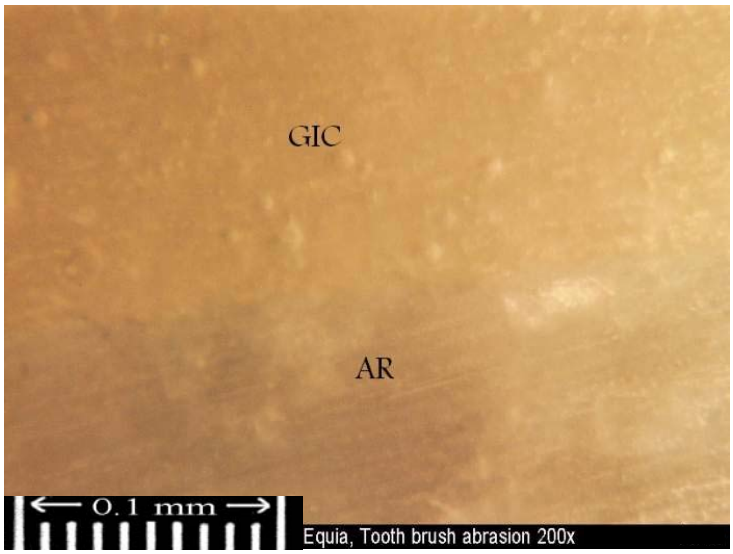

FIGURE 16. Cross-sectional micrograph of EQUIA specimen after toothbrush abrasion test. (Specimen 2) (GIC= glass ionomer; AR= acrylic resin.)

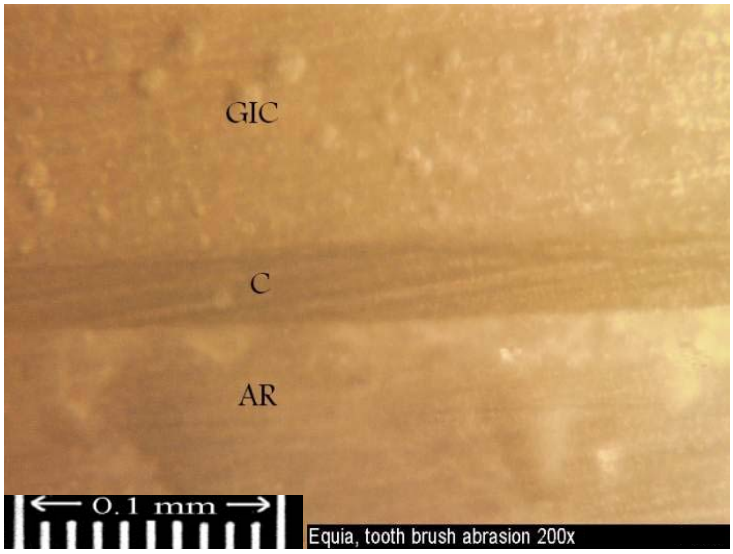

FIGURE 17. Cross-sectional micrograph of EQUIA specimen before toothbrush abrasion test. (Specimen 3) (GIC= glass ionomer; AR= acrylic resin; C= nano-filled resin coat)

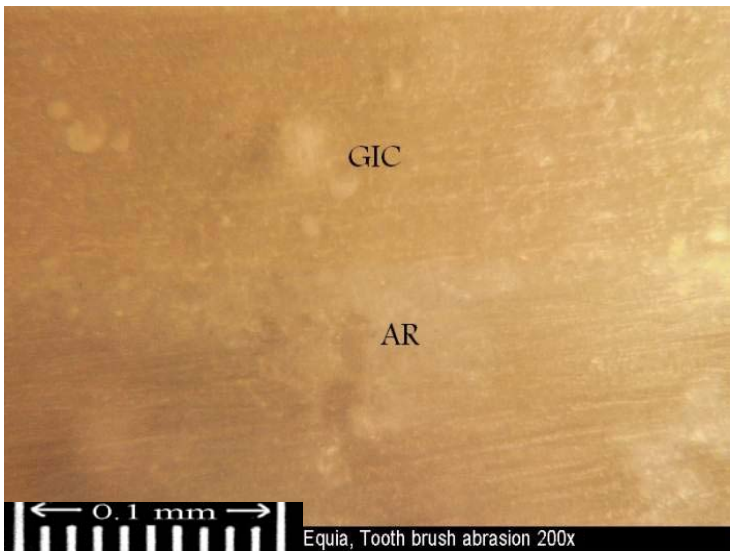

FIGURE 18. Cross-sectional micrograph of EQUIA specimen after toothbrush abrasion test. (Specimen 3) (GIC= glass ionomer; AR= acrylic resin)

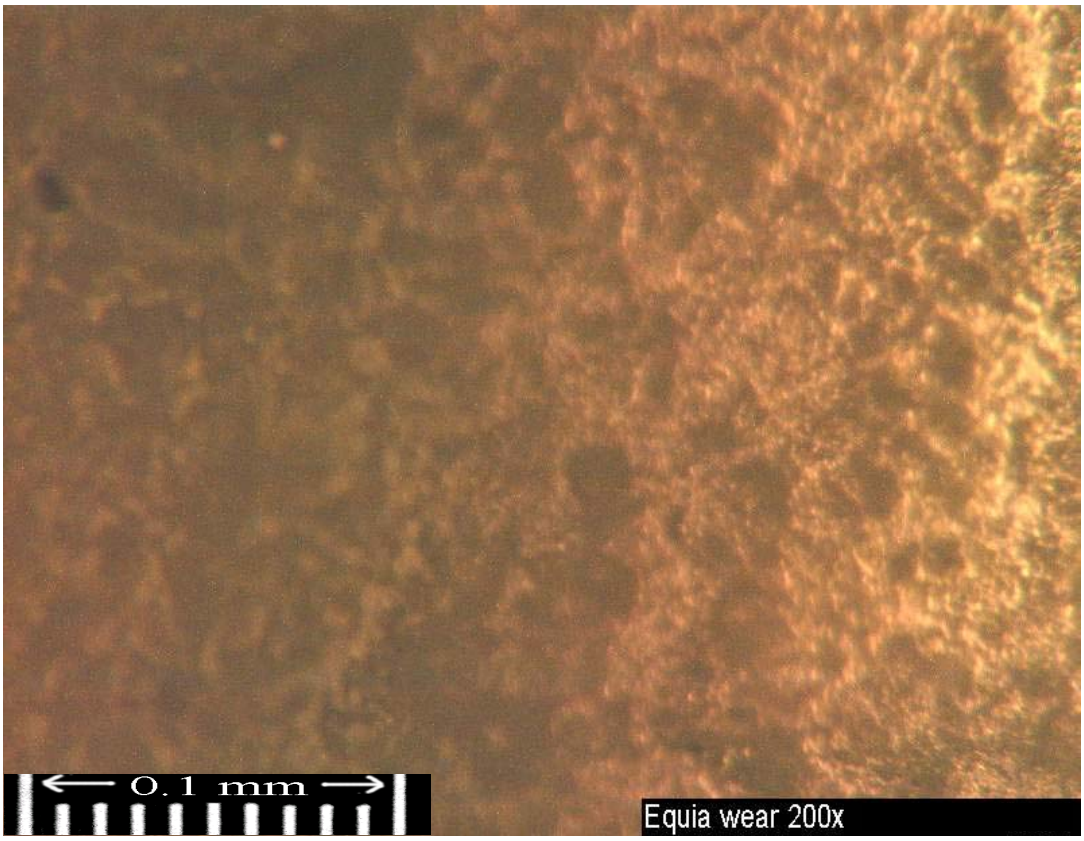

FIGURE 19. Micrograph of worn EQUIA surface after three-body Alabama wear test.

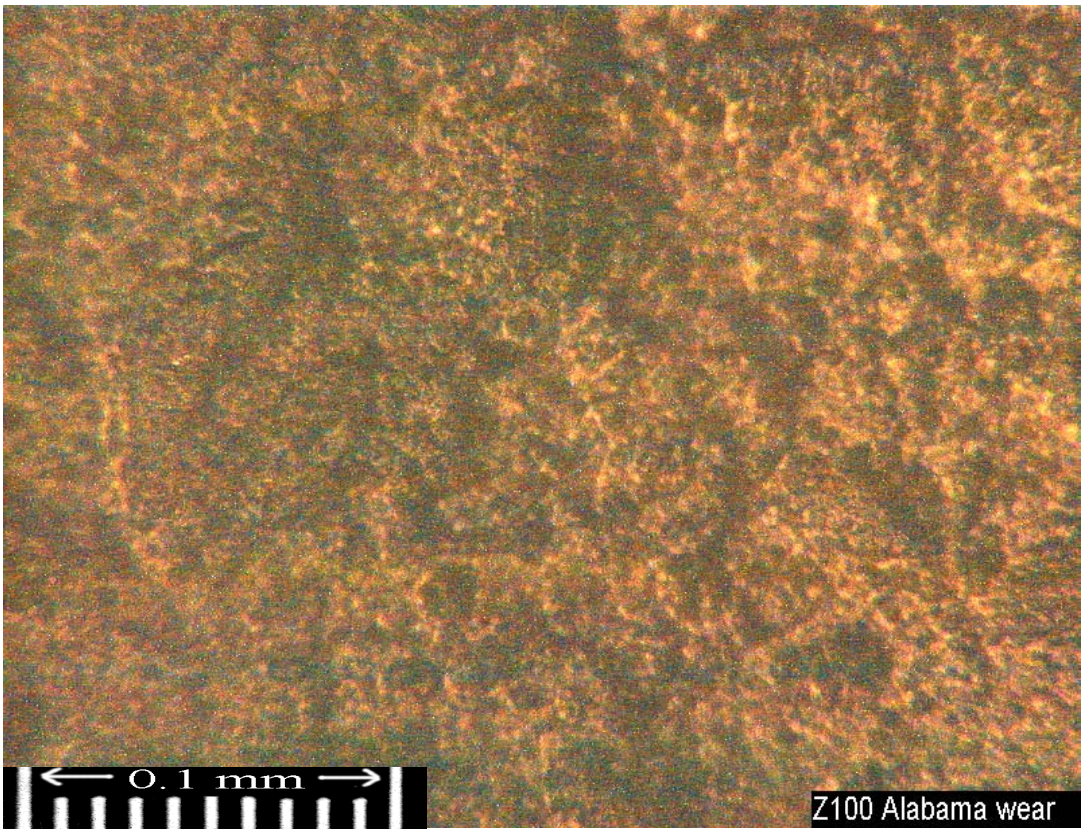

FIGURE 20. Micrograph of worn Z100 surface after three-body Alabama wear test.

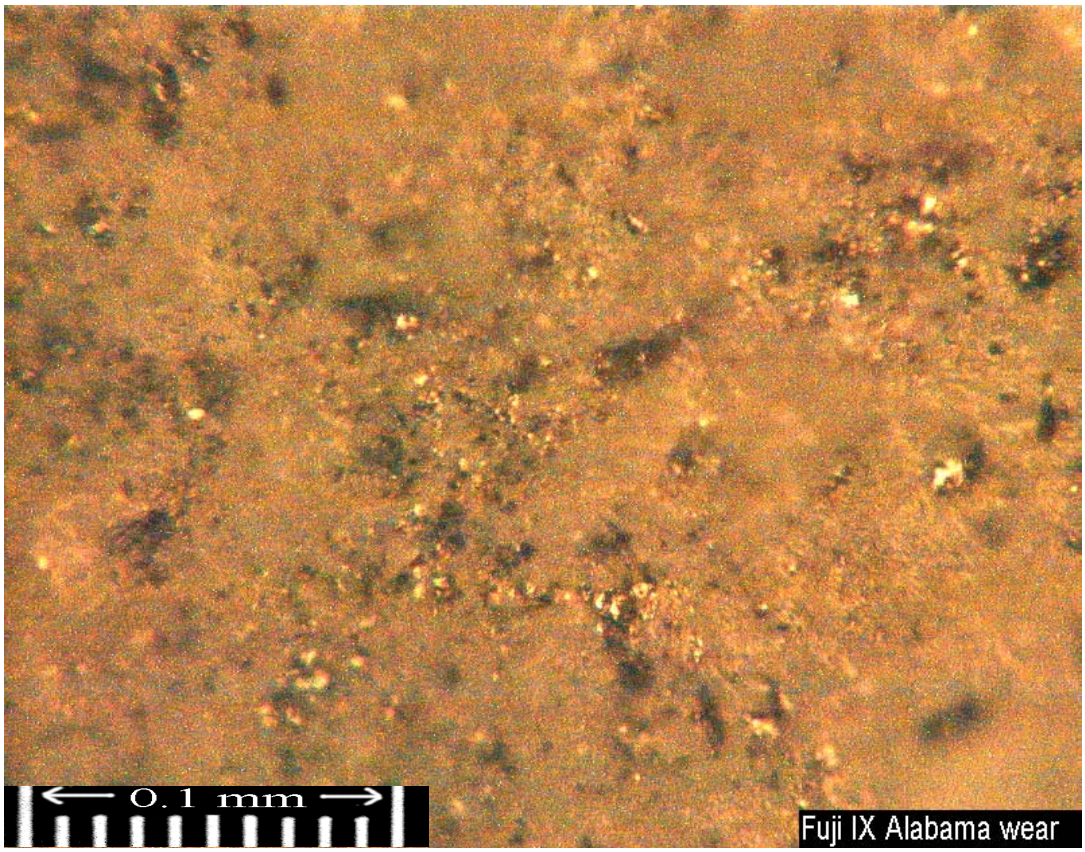

FIGURE 21. Micrograph of worn Fuji IX GP Extra surface after three-body Alabama wear test.

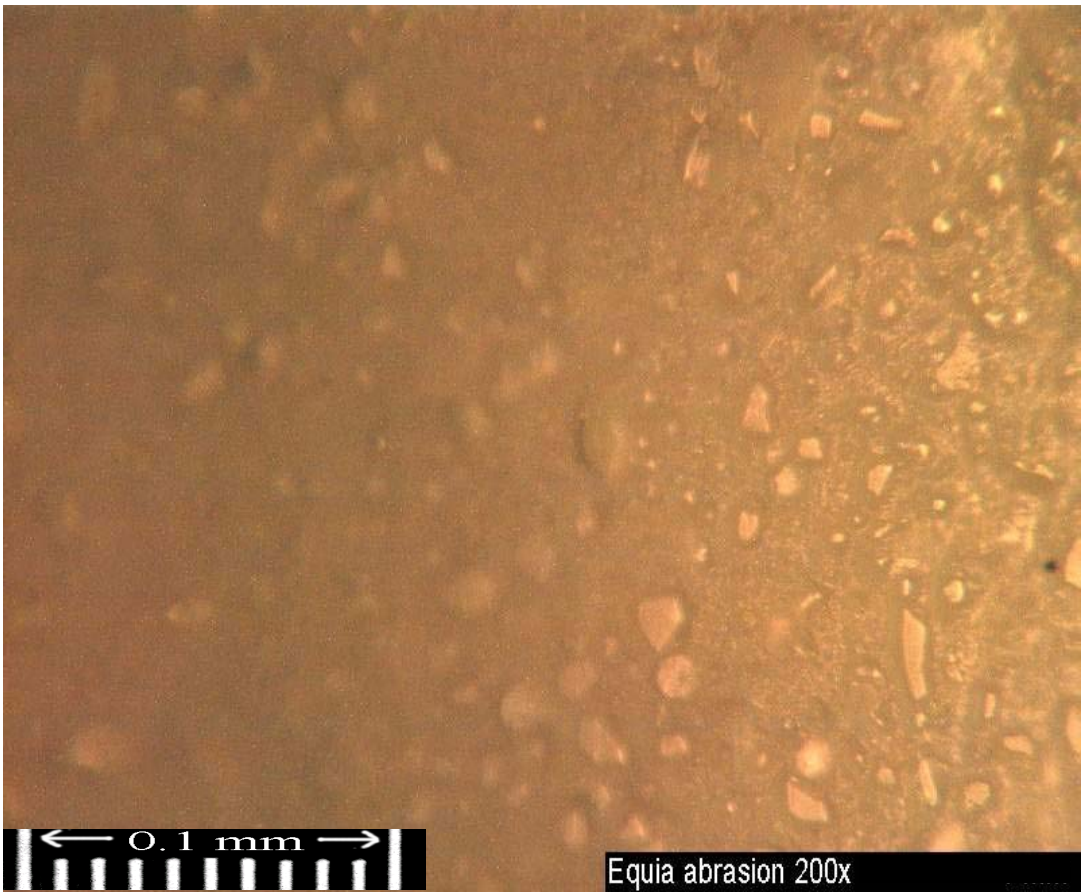

FIGURE 22. Micrograph of worn EQUIA surface after toothbrush abrasion test.

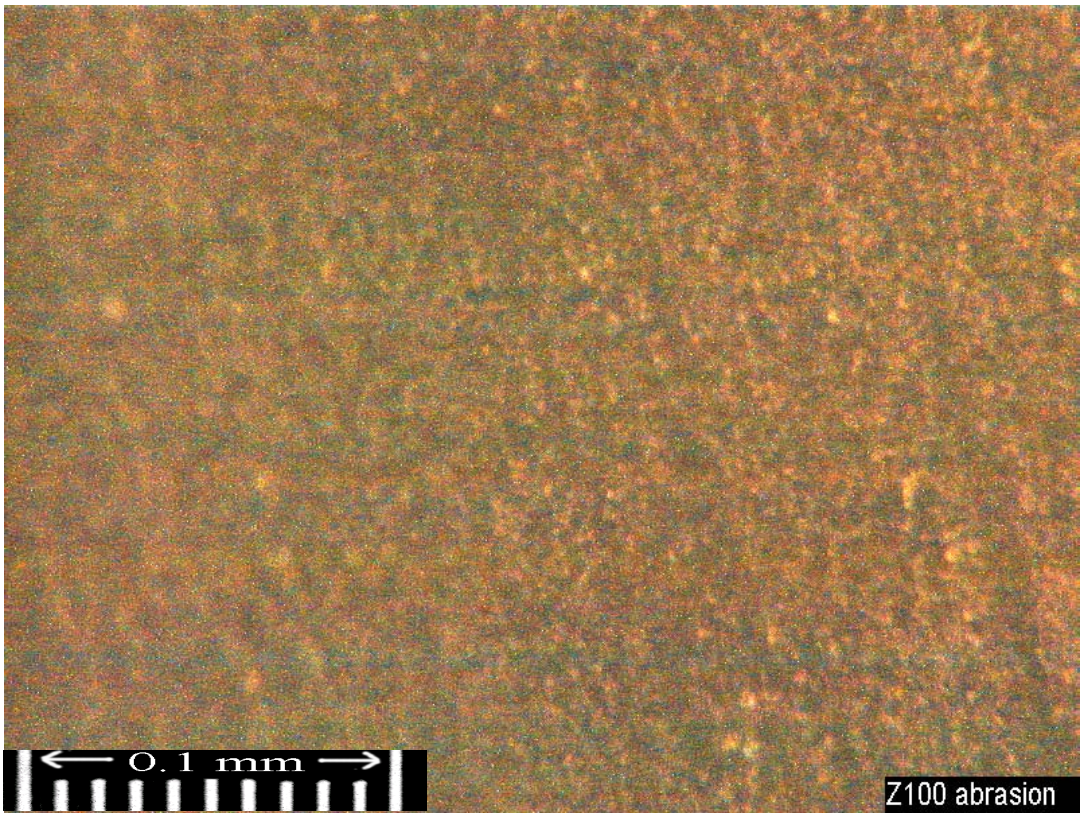

FIGURE 23. Micrograph of worn Z100 surface after toothbrush abrasion test.

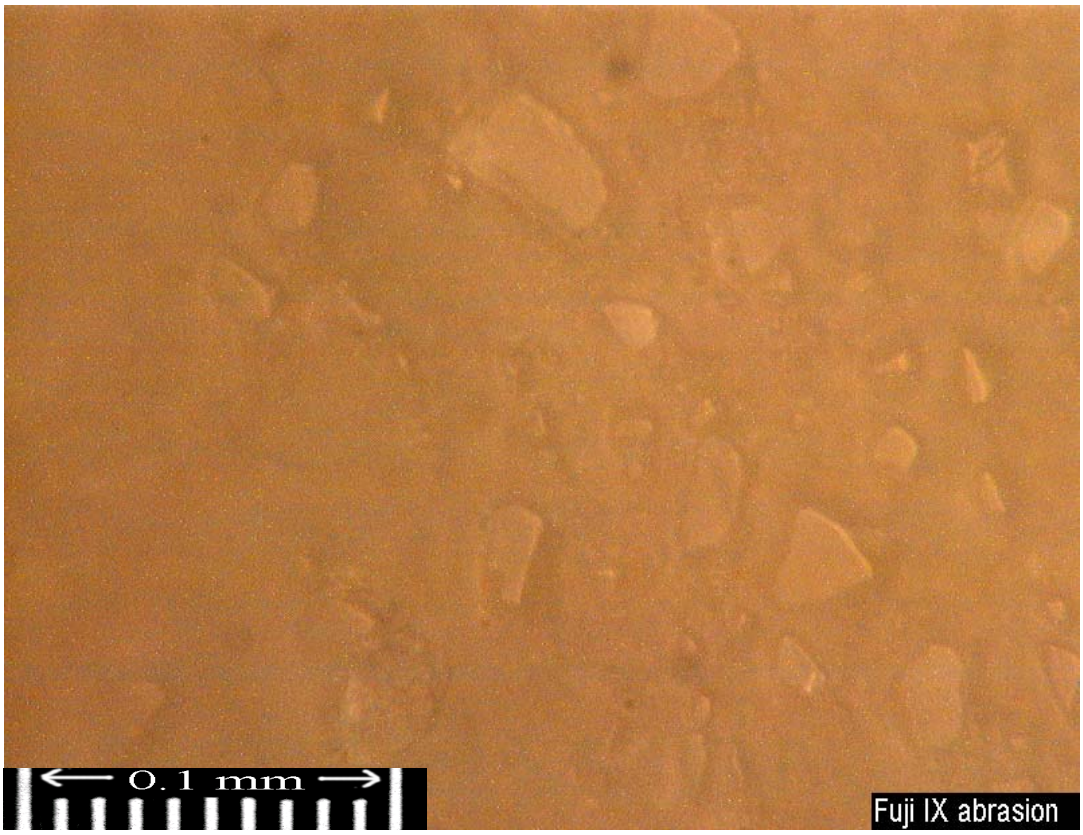

FIGURE 24. Micrograph of worn Fuji IX GP Extra surface after toothbrush abrasion test.

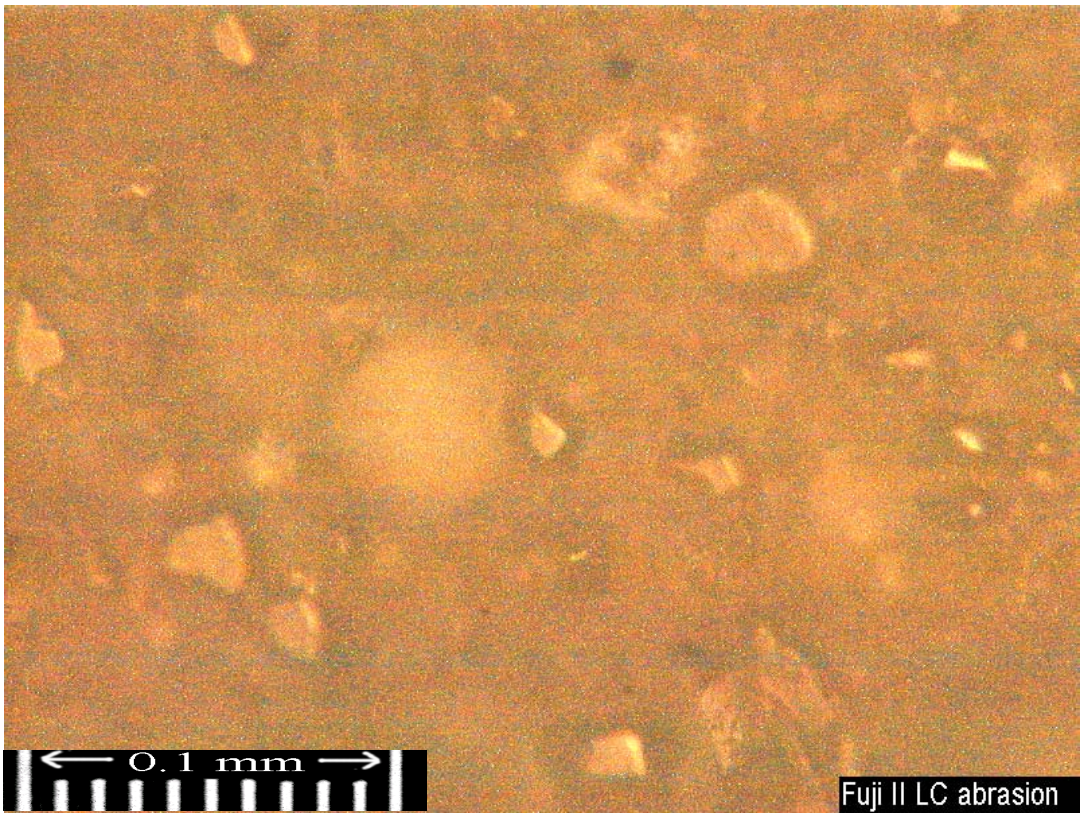

FIGURE 25. Micrograph of worn Fuji II LC surface after toothbrush abrasion test.

DISCUSSION

GIC restorations have several advantages compared with other dental materials. These advantages make them suitable for restoring primary teeth. Clinical studies on class V cavities restored with GICs and clinical studies of the Atraumatic Restorative Treatment (ART) using glass ionomer as the restorative material show promising results.[21, 57, 58] The weak mechanical properties of GICs make them inferior to other direct restorative materials for restoring teeth in high-stress areas. Manufacturers have tried several methods to improve the mechanical properties of GICs. One result of these methods was the introduction of the new glass ionomer system called EQUIA by GC America. The manufacturer claims this material has improved mechanical properties and can be used as a replacement for amalgam and composite resin when restoring posterior teeth. The objective of this study was to evaluate the wear resistance of this new material and compare it with the wear resistance of other restorative materials.

THREE-BODY ALABAMA WEAR

The current study used a three-body Alabama wear testing machine to simulate the natural wear process that happens in the oral cavity. This method has been used in several studies to evaluate wear resistance of several materials. Some studies have used enamel as an antagonist to assess the wear resistance of enamel against different materials in the oral cavity.[37] Others have used different types of materials as antagonists in comparing wear resistance.[38, 40]

The results of this study showed that wear resistance of conventional GIC is significantly inferior to composite resin. This finding is in agreement with the results from previous studies.[1, 2] When a nano-filled resin coating is applied to the finished surface of the conventional GIC, the wear resistance is improved and becomes comparable to the wear resistance of composite resin, which means the coat protects the surface of the restoration and increases the wear resistance.

After 400,000 wear cycles in the three-body Alabama wear machine, cross-sectional micrographs of EQUIA specimens (Figure 7 through Figure 12) showed the resin coat is still present in the worn area, although it gets thinner compared with unworn areas. After testing, figures showed that resin coating lost about 15 um to 20 μm of its thickness, which is about 35 percent to 50 percent of its original thickness.

Studies show that humans perform 250,000 chewing cycles per year.[59, 60] According to the results of the present study, the resin coat is expected to stay on the surface of the restoration for more than a year-and-a-half. This expectation might not correlate with everyday chewing because a polyacetyl material was used as the antagonist in this study, which is different from the enamel in natural teeth. It was difficult to use enamel as an antagonist in this study because of the difficulty in preparing enamel specimens to the dimensions used in the three-body Alabama wear machine.

Examination of the worn surface under the metallograph showed two different wear patterns between the three-body Alabama wear and the toothbrush abrasion. In the toothbrush abrasion test (Figure 22 to Figure 25), the wear appears to primarily affect the matrix of the cement while the glass particles appear to be in place and not affected. In the three-body Alabama wear test (Figure 19 to Figure 21) the micrograph shows that the

wear affects both the matrix and the filler particles. It shows destroyed and plucked filler particles.

Resin-modified GICs were the least wear-resistant among the tested materials. This finding is in agreement with results of previous studies.[7, 45]

TOOTHBRUSH ABRASION

The toothbrush abrasion test has been used in several studies to evaluate the ability of materials to resist the abrasion caused by tooth brushing.[36, 39] The machine simulates the tooth brushing technique by moving a toothbrush head back and forth on the surface of the specimen in the presence of an abrasive medium. Although the principle mechanism of the test is similar in different studies, they differ in the time, frequency, and applied load. This makes it difficult to compare the results from different studies. In this study, the toothbrush abrasion results showed no difference in abrasion resistance between the composite resin, the coated GIC, and the uncoated GIC.

In contrast to the three-body Alabama wear test, cross-sectional micrographs (Figure 13 to Figure 18) of the EQUIA specimens, after the toothbrush abrasion test, showed complete wearing off of the coat and exposure of the underlining glass ionomer surface. A possible explanation for this is the difference in wear mechanism between the two tests. In the three-body Alabama wear test, the hard filler particles in the coating provide some protection for the matrix against the hard flat surface of the antagonist. In the toothbrush abrasion test, the abrasiveness process affected mainly the matrix of the coating and resulted in complete wearing off of the coat. This could explain why there is no difference in abrasion resistance between EQUIA and Fuji IX GP Extra, when tested in the toothbrush abrasion test.

According to a study by Kanter, the number of strokes needed to simulate one year of average human brushing is about 4320 strokes.[61] In this study, EQUIA showed comparable wear resistance values to composite resin when tested in toothbrush abrasion testing after 20,400 brushing strokes. This simulates more than four-and-a-half years of tooth brushing. Although this is considered an acceptable result, it is important to note that it is an estimate. It is difficult to calculate the actual number of strokes for a specific period of time, because technique differs from one person to another. In addition to that, the amount of the load applied to the toothbrush head during brushing is different from one person to another.

MICROHARDNESS

The microhardness of the resin composite in the present study was the highest compared with the other tested materials. This result is in agreement with results from a previous study.[1]

In restorative dental resin composite materials, fillers are considered the strongest phase. Their primary purpose is to strengthen composite and to reduce the amount of weak matrix material, resulting in increased hardness, strength, and decreased wear. Previous studies show that increasing the filler size and content improves the mechanical properties of the material.[62, 63] In this study, the microhardness test results showed that EQUIA had lower microhardness values compared with the composite resin and the uncoated GIC. These findings were expected because, in the case of EQUIA, the microhardness values come from the coating on the surface of the glass ionomer, which has less filler than the glass ionomer materials.

Several limitations in this study need to be addressed in future studies before considering EQUIA as an acceptable final restorative material for posterior teeth. In the present study, EQUIA showed high wear-resistance values comparable to resin composite. However, other mechanical properties including fracture toughness and strength need to be taken into consideration for this material. Further studies are necessary to evaluate the effect of the coating procedure on the physical properties of this material.

Another limitation in the present study is the short testing time. Although EQUIA showed good wear-resistance values within the time line of this study, the material needs to be evaluated for a longer period of time and under different loads to assess its performance compared with other restorative materials. Another limitation that makes the results of this study less clinically relevant is the type of antagonist used in the three-body Alabama wear test. In this study, polyacetyl sliders were used as an antagonist, which have different mechanical properties than hydroxyapatite in dental enamel.

In addition to the above-mentioned limitations, the design of the present study has a lot of variables that could affect the results. These variables include the softness of the toothbrush head, the abrasiveness of the abrasive medium, the frequency of brushing cycles, and the applied load. Changing these variables may affect the results of this study either in a positive or a negative way.

SUMMARY AND CONCLUSION

The objective of this study was to measure the toothbrush abrasion, wear resistance, and microhardness of a newly introduced high-strength GIC coated with nano-filled resin and compare it with other restorative materials. Four different materials were chosen and compared in this study, including composite resin, resin modified GIC, conventional high-strength GIC, and conventional high-strength GIC coated with nano-filled resin. The three-body Alabama wear test, the toothbrush abrasion test, and the microhardness test were used to test the materials.

From the data collected, the results can be summarized as follows:

1) EQUIA has wear resistance values comparable to composite resin and higher than values for conventional GIC.
2) Resin-modified GICs showed the highest wear among all tested materials.

In conclusion, the present study showed that coating the surface of a glass ionomer material with a nano-filled resin results in increasing the wear resistance of the material and making it comparable to a resin composite material within the limitations of this study.

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ABSTRACT

IN-VITRO WEAR AND HARDNESS OF NEW CONVENTIONAL GLASS IONOMER CEMENT COATED WITH NANO-FILLED RESIN

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Background: Since the introduction of glass ionomer cements (GICs) in the 1970s, many attempts have been made to improve them and expand their application in restorative dentistry. Recently, GC America introduced a new glass ionomer restorative system called EQUIA. The manufacturer claims that this material has improved wear resistance by coating the surface of high-strength GIC with a nano-filled resin coating. Objective: The objective of this study was to measure the wear resistance and hardness of EQUIA and to compare it to other current restorative materials. Materials and Methods: Four different materials were used in this study: EQUIA, Fuji IX GP Extra, Fuji II LC and Z-100. Six specimens of each material were made and then tested in a toothbrush abrasion machine for 20,400 cycles, after which the amount of volume loss was

calculated. Eight specimens of each material were made and tested in a three-body Alabama wear testing machine under a load of 75 N for 400,000 cycles. Four surface profiles were obtained from each specimen and volume loss was calculated using computer software. Five specimens of each material were made and Knoop microhardness was determined by using the mean of the three values from the top surface of the specimen. Results of each test were collected and compared with the other materials using one-way analysis of variance (ANOVA) at a significance level of 0.05. Results: Wear-resistance results showed that EQUIA has wear-resistance values comparable to composite resin and higher values than those for the high-strength GIC. The results also showed that Fuji II LC had the highest wear among all tested materials. Microhardness results showed that EQUIA has significantly lower microhardness than Fuji IX GP Extra and Z-100. Conclusion: Based on the results of the present study, it can be concluded that coating the surface of glass ionomer restorations with a nano-filled resin coat results in increasing the wear resistance and decreasing the microhardness of the material. Within the limitations of this study, EQUIA has comparable wear resistance to composite resin.

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