©*Operative Dentistry*, 2014, **39-3,** E109-E117

Influence of Chemical Degradation on the Surface Properties of Nano Restorative Materials

AB de Paula • SBP de Fúcio • RCB Alonso
GMB Ambrosano • RM Puppin-Rontani

Clinical Relevance

This study demonstrated that the incorporation of nanoparticles does not improve the surface properties of the ionomeric cements when exposed to simulated dietary solutions. However, the nanofilled composite resin was more resistant to chemical degradation.

SUMMARY

Objectives: The aim of this *in vitro* study was to investigate the effect of chemical degradation on the surface roughness (R_a) and hardness (Knoop hardness number [KHN]) of nano restorative materials.

Methods: Disc-shaped specimens (5-mm diameter; 2-mm thick) of Filtek Z350 and TPH Spectrum composites and the Vitremer and Ketac Nano light-curing glass ionomer cements were prepared according to the manufacturers' instructions. After 24 hours, polishing procedures were performed and initial measurements of R_a and KHN were taken. The specimens were divided into 12 groups (n=10) according to material and storage media: artificial saliva, orange juice, and Coca-Cola. After 30 days of storage, the specimens were reevaluated for R_a and KHN. The pH values of the storage media were measured weekly. Data were tested for significant differences by repeated-measures three-way analysis of variance and Tukey tests ($p<0.05$).

Results: Composites were found to present lower roughness values and higher hardness values than the ionomeric materials under all storage conditions. After degradation, the KHN of all experimental samples decreased significantly, while the R_a of the ionomeric materials increased, depending on the media, with a markedly negative impact of Coca-Cola and orange juice. There was no difference among the storage media for Filtek Z350 with

Andréia Bolzan de Paula, DDS, MS, PhD, Restorative Dentistry Department, Dental Materials Area, Piracicaba Dental School, University of Campinas, Piracicaba, São Paulo, Brazil

Suzana Beatriz Portugal de Fúcio, DDS, MS, PhD, Restorative Dentistry Department, Dental Materials Area, Piracicaba Dental School, University of Campinas, Piracicaba, São Paulo, Brazil

Gláucia Maria Bovi Ambrosano, DDS, MS, PhD, Social and Public Health Department, Piracicaba Dental School, University of Campinas, Piracicaba, São Paulo, Brazil

Roberta Caroline Brusch Alonso, DDS, MS, PhD, Biomaterials Department, Bandeirante University of São Paulo-UNIBAN, São Paulo, Brazil

*Regina Maria Puppin-Rontani, DDS, MS, PhD, Pediatric Dentistry Department, Piracicaba Dental School, University of Campinas, Piracicaba, São Paulo, Brazil

*Corresponding author: UNICAMP, Av. Limeira, 901, 13414-900, Piracicaba, SP, Brazil; e-mail: rmpuppin@fop.unicamp.br

DOI: 10.2341/12-340

Table 1: *Materials Used in This Study*

Material	Composition	Mean Filler Size, μm	Manufacturer/Batch No.
Ketac Nano (3M ESPE)	Paste A: silane-treated glass, silane-treated zirconia oxide silica, polyethylene glycol dimethacrylate (5%-15%), silane-treated silica, HEMA, Bis-GMA (<5%), TEGDMA (<5%), HEMA (1%-10%) Paste B: silane-treated ceramic, silane-treated silica, copolymer of acrylic and itaconic acids, HEMA (1%-10%)	1 μm (cluster) 5-25nm (nanofiller) <3.0 μm (glass)	3M/ESPE M3M3
Vitremer™ (3M ESPE)	Powder: fluoroaluminosilicate glass; redox system Liquid: aqueous solution of a modified polyalkenoic acid, HEMA (15%-20%)	3.0 μm	3M/ESPE P: 6LP L: 6FH
Filtek™ Z350 (3M ESPE)	58%-60 vol% (78.5 wt%) combination of aggregated zirconia/sílica cluster filler with primary particles size of 5-20 nm and nonagglomerated 20-nm silica filler, Bis-EMA, Bis-GMA; UDMA; TEGDMA	5-20 nm 0.6-1.4 μm (clusters)	3M/ESPE 8NU
TPH Spectrum™ (Dentsply)	Polymer matrix: Bis-GMA, Bis-EMA, and TEGDMA; filler: 57 vol% of Ba-Al-borosilicate glass and coloidal sílica with mean particle size of 0.8 μm	4.4 μm	Dentsply Ind. E Com Ltda L797977

Abbreviations: Bis-EMA, ethoxylated bisphenol-A dimethacrylate; Bis-GMA, bisphenol glycidyl methacrylate; HEMA, 2-hydroxyethyl methacrylate; TEGDMA, triethylene glycol dimethacrylate; UDMA, urethane dimethacrylate.

regard to the KHN values. Nanofillers did not show any influence on the roughness and hardness of resin-modified glass ionomer cements and resin composites concerning their degradation resistance.

INTRODUCTION

The ability of restorative dental materials to withstand occlusal forces and exposure to various substances in the mouth is an important requirement for their clinical performance over a considerable period of time. The chemical factors known to cause deleterious effects include low pH due to cariogenic biofilm,[1] consumption of acidic drinks or food stuffs,[2,3] and the action of enzymes,[4] which can soften and roughen the outermost layers of restorative materials. Glass-ionomer cement degradation is a complex process of extracting metal cations from the cement matrix and incorporated glass particles,[5] resulting from fluid uptake by the matrix and its solubility. Still, the highly hydrophilic hydroxyethyl methacrylate (HEMA) present in resin-modified glass ionomer (RMGI) makes this material also susceptible to the disintegration of its matrix, in a variable performance heavily dependent on the resin matrix composition and polymerization reactions.[6]

The application of nanotechnology to composite resins was introduced by Filtek Supreme (3M-ESPE, St. Paul, MN, USA), which contains a unique combination of nanofillers (5-75 nm) and nanoclusters embedded in an organic polymer matrix. In addition to improved optical properties, nano materials present better mechanical behavior,[7] since the particle size increased the nanofiller load in the restorative materials.[8] Some studies have shown that nano composites presented higher surface hardness values and lower brushing abrasive wear than did microfilled and hybrid composites.[9,10] Recently, a new RMGI was introduced for operative dentistry. Ketac Nano, a light-curing nano-ionomer (3M ESPE), includes fluoroaluminosilicate glass, nanofillers, and nanofiller clusters combined with HEMA, bisphenol glycidyl methacrylate (Bis-GMA), and triethylene glycol dimethacrylate (TEGDMA) as resin monomers, (3M ESPE, internal data).[11]

According to the manufacturer, Ketac Nano presents mechanical properties such as compressive and flexural strength, diametral tensile, and wear resistance greater than most other glass ionomers tested.[11] Still, the new material showed high polish and surface roughness after tooth brushing, similar to composite.[11] Some studies *in vitro* have shown that the inclusion of nanotechnology in Ketac Nano really provided an ionomeric material with better properties under chemical (biofilm interaction and pH cycling) and mechanical challenge,[12,13] applications of acidulated phosphate fluoride,[14] and after polishing procedures.[15] Nevertheless, a randomized clinical trial with one-year results showed enamel marginal deficiencies and color mismatch more prevalent for Ketac Nano.[16]

Whereas the resin matrix of this material has also undergone modifications (additional monomers to HEMA) and this material is indicated for erosion lesions,[11] often caused by frequent ingestion of acidic foods and drinks, it would be important to evaluate the effects of different beverages on the surface roughness and hardness of nano restorative materi-

Table 2: *Main Ingredients in the Storage Solutions Studied*

Food/Drink	Main Ingredients	pH
Coca-Cola	Carbonated water, sugar, caramel color, phosphoric acid, natural flavors, caffeine	2.49
Orange juice (Minute Maid)	Water, orange juice, sugar, citric acid, natural flavor, and antioxidant ascorbic acid	3.23
Artificial saliva	Calcium (0.1169 g of calcium hydroxide/L of deionized water); 0.9 mM of phosphorus and potassium (0.1225 g potassium phosphate monobasic/L of deionized water); 20 mM TRIS buffer (2.4280 g TRIS buffer/L deionized water)	7

als (a nano-ionomer and a nanocomposite), when compared with another resin-modified glass ionomer cement and composite.

MATERIALS AND METHODS

Specimen Preparation and Initial Analysis

Four different types of tooth-colored restorative materials were used in this present study (Table 1): two resin-modified glass ionomers (Vitremer and Ketac Nano, 3M ESPE) and two composites (Filtek Z350 [3M ESPE] and TPH Spectrum [Dentsply Ind & Com Ltda, Petropolis, Brazil]). Thirty specimens of each material were prepared according to the manufacturers' instructions under aseptic conditions and inserted into plastic molds with internal dimensions of 5-mm diameter and 2-mm thick. The surface of each specimen was covered by a polyester strip and pressed flat by a glass slab. The top surface of all materials was cured according to manufacturers' cure times using an Elipar Trilight curing light unit (3M ESPE), with a mean intensity of approximately 800 mW/cm^2 as checked with a curing light meter (Hilux Dental Curing Light Meter, Benliglu Dental Inc, Turkey). The surface of Vitremer was protected with finishing gloss (3M ESPE). All specimens were maintained at 100% relative humidity and 37°C for 24 hours. The surfaces were then wet-ground with water-proofed silicon carbide discs of decreasing abrasiveness (600, 1200, and 2000) and ultrasonically cleaned (Ultrasonic Cleaner, model USC1400, Unique Co, São Paulo, Brazil) in distilled water for 10 minutes to remove polishing debris. Then, the specimens were randomly distributed into three groups (n=10) according to the test storage medium: artificial saliva (control), orange juice, and Coca-Cola (Table 2).

Before chemical degradation in acidic drinks, all specimens were analyzed for surface roughness and Knoop hardness. For surface roughness testing, the specimens were analyzed using a Surfcorder SE1700 instrument (Kosaka Corp, Tokyo, Japan), with cutoff length of 0.25 mm, at a tracing speed of 0.1 mm/s. The mean surface roughness values (R_a, mm) of each specimen were obtained from three successive measurements of the center of each disk in different directions (total length analyzed of 3.750 mm). Next, hardness tests were carried out with a hardness tester (Shimatzu, Tokyo, Japan) using a Knoop indenter and a load of 50 g, with a dwell time of 15 seconds. Three readings were taken for each specimen, and the mean KHN was calculated for each disk. Then, 10 specimens with surface roughness and hardness values previously determined were distributed for each group of different storage solutions (n=10).

Chemical Degradation: Storage in Acidic Drinks

All specimens were immersed individually in 4 mL of storage solutions, Coca-Cola (pH 2.49), orange juice (pH 3.23), and artificial saliva (pH 7), for 30 days.[17] The solutions were changed weekly and pH tested using a portable pH meter (Orion Model 420A, Analyzer, São Paulo, Brazil). In all cases, the pH electrodes were calibrated immediately prior to use with the aid of standard buffer solutions at pH 4.0 and 7.0.

At the end of the storage period, the specimens were ultrasonically washed for 10 min, dried, and revaluated for roughness and hardness. A representative specimen from each group was also observed under scanning electron microscopy (model Jeol JSM 5600 LV, Tokyo, Japan) to illustrate the effect of the erosive challenge on the materials. Additional specimens from each material were made to serve as a baseline to compare baseline surfaces to chemically degraded surfaces.

Statistical Analysis

Data were evaluated using the PROC LAB from SAS to check the equality of variances and confirm a normal distribution. Hardness and roughness data were submitted to repeated-measures three-way analysis of variance (ANOVA) and Tukey test with a significance limit of 5%. Hardness data were transformed using root square to allow the use of ANOVA.

Table 3: *Mean (SD) Surface Roughness Values (μm) of Restorative Materials Submitted to Different Storage Solutions*[a]

Erosion	Material	Storage Solution		
		Artificial Saliva	Coca-Cola	Orange Juice
Before	Filtek Z350	0.14 (0.07)Ab	0.13 (0.03)Ab	0.13 (0.03)Ab
	TPH Spectrum	0.18 (0.04)Ab	0.18 (0.03)Ab	0.20 (0.04)Ab
	Ketac Nano	0.33 (0.12)Aa	0.37 (0.13)*Aa	0.31 (0.11)*Aa
	Vitremer	0.47 (0.19)Aa	0.39 (0.17)*Aa	0.34 (0.09)*Aa
After	Filtek Z350	0.11 (0.01)Ab	0.11 (0.01)Ab	0.11 (0.01)Ab
	TPH Spectrum	0.17 (0.02)Ab	0.17 (0.05)Ab	0.19 (0.05)Ab
	Ketac Nano	0.32 (0.14)Aa	0.48 (0.16)Aa	0.40 (0.11)Aa
	Vitremer	0.40 (0.09)Aa	0.48 (0.16)Aa	0.49 (0.15)Aa

[a] *Capital letters indicate comparison among storage solutions (horizontal). Lowercase letters demonstrate comparison among materials (vertical) within each storage solution and each erosion condition (before or after). Asterisks represent statistically significant difference between erosion effect (before × after). Groups denoted by the same letter/symbol represent no significant difference ($p>0.05$).*

RESULTS

There was a significant interaction between the factors "materials" and "degradation effect" (before × after; $p=0.0439$) and between "storage solution" and "degradation effect" ($p=0.0074$). A significant interaction was not observed between "materials" and "storage solution" ($p=0.4733$) and among the three factors ($p=0.0699$). The means and standard deviations of surface roughness of each material under different storage conditions are displayed in Table 3. There was no significant difference among storage solutions (saliva/orange juice/Coca-Cola; $p=0.2010$) and between degradation effect (before/after; $p=0.2251$); however, there were statistical differences among the materials studied ($p<0.0001$).

Regardless of the storage solution, both composites (Filtek Z350 and TPH Spectrum) presented similar roughness values ($p>0.05$) and significantly lower roughness values than the ionomers, both before and after the chemical challenges. There was no statistical difference in roughness values between Ketac Nano and Vitremer in all storage conditions. In addition, when different storage solutions were compared for each material after chemical challenge, no statistically significant difference among them was observed. With regard to the degradation effects on the surface of each material, the exposure to acidic drinks (orange juice and Coca-Cola) resulted in chemical degradation (ie, significantly higher roughness values for both of the ionomeric materials tested). Artificial saliva did not produce any difference in the roughness results for all materials. Composite surfaces were not rough when kept in any storage solution evaluated.

Table 4 presents the mean and standard deviations of the Knoop hardness values of each material under the evaluated storage conditions. There was a significant interaction between the factors "materials" and "storage solution" ($p=0.0009$), "materials" and "degradation effect" ($p<0.0001$), and "storage solution" and "degradation effect" ($p<0.0001$), as well as among the three factors ($p=0.0022$). In addition, there was a significant difference among the materials ($p<0.0001$), storage solutions (saliva/juice/Coca-Cola; $p<0.0001$), and degradation effect (before/after; $p<0.0001$).

Before degradation in acidic beverages, both of the composites (Filtek Z350 and TPH Spectrum) presented similar and significantly higher hardness values than the ionomers evaluated, which presented similar values between them. Regarding the degradation effects, the exposure to any storage solution produced statistically lower hardness values for all materials tested. There was also an influence of the storage solution found for each material: the acidic beverages (Coca-Cola and orange juice) were more aggressive than artificial saliva for Vitremer and Keta Nano. Orange juice was also very detrimental to TPH Spectrum, while no difference was observed among the solutions for Filtek Z350. In addition, the composites presented significantly higher hardness values than did the ionomeric materials after chemical degradation by artificial saliva and Coca-Cola. However, after orange juice storage, Filtek Z350 showed the highest hardness values, followed by TPH Spectrum and, finally, by both ionomeric materials.

DISCUSSION

This current study evaluated the effects of acidic beverages on the surface roughness and hardness of nano and conventional restorative materials. Three storage media were selected: orange juice and Coca-Cola, due to their potential to cause chemical

Table 4: *Mean (SD) Knoop Hardness Number (KHN) of Restorative Materials Submitted to Different Storage Solutions*[a]

Erosion	Materials	Storage Solutions		
		Artificial Saliva	Coca-Cola	Orange Juice
Before	Filtek Z350	78.11 (8.55)*Aa	84.17 (10.79)*Aa	82.06 (12.31)*Aa
	TPH Spectrum	81.84 (11.15)*Aa	79.93 (9.11)*Aa	79.43 (10.97)*Aa
	Ketac Nano	41.16 (5.29)*Ab	39.65 (5.79)*Ab	39.64 (6.83)*Ab
	Vitremer	39.12 (4.53)*Ab	40.31 (7.83)*Ab	39.41 (8.43)*Ab
After	Filtek Z350	65.33 (5.80)Aa	57.97 (6.60)Aa	65.13 (7.46)Aa
	TPH Spectrum	64.30 (5.22)Aa	52.35 (5.76)ABa	42.85 (4.96)Bb
	Ketac Nano	27.38 (4.18)Ab	18.92 (2.18)Bb	15.53 (2.69)Bc
	Vitremer	28.91 (2.76)Ab	16.29 (4.19)Bb	19.45 (4.27)Bc

[a] *Capital letters indicate comparison among storage solutions (horizontal). Lowercase letters demonstrate comparison among materials (vertical) within each storage solution and each erosion condition (before or after). Asterisks represent statistically significant difference between erosion effect (before × after). Groups denoted by the same letter/symbol represent no significant difference ($p>0.05$).*

degradation, and artificial saliva, as a positive control. Coca-Cola contains phosphoric acid and has low titratability. Orange juice contains citric acid, which has a high titratability and buffering capacity.[18] Overall, both storage solutions caused a significant increase in the surface roughness values for resin-modified glass ionomers cements (RMGIC) studied and a significant decrease in hardness for all materials, confirming their potential to degrade resin-based restorative materials.

Before the degradation process, higher roughness values were observed for RMGIC when compared with composite resins. The differences observed at the baseline conditions among materials regarding their means of surface roughness values are mainly related to differences in their filler particle size, shape, volume, and distribution and their interaction with the organic matrix, allowing better polishing characteristics for the composites.[19] Still, the handling of two pastes or powder/liquid can trap air in the ionomeric material structure, resulting in surface bubbles and exposure of porosities after finishing/polishing procedures.

Prior to acidic challenge, similar roughness values between the nanofilled and conventional materials were observed, for both the composite and ionomer groups. However, Mitra and Holmes[7] and Cavalcante and others[20] demonstrated that nanofilled composites presented lower roughness values and better polishing characteristics than did hybrid composites because of the presence of nanofillers. The same behavior was observed for Ketac N100 compared with other ionomeric materials.[15] Nevertheless, the resinous matrix of the materials used in this study was not probably totally removed by the initial finishing/polishing procedures, leaving a thin matrix layer over the fillers (as observed in micrographs), which interfered in the superficial nanofillers' presence and exposition.

Composite resins and RMGICs performed differently with regard to their surface roughness when evaluated for the 30-day chemical degradation period. The chemical challenge caused no effects on the roughness values for both composites. The ethoxylated version of the Bis-GMA (Bis-EMA) existing in the composition of Filtek Z350 and TPH Spectrum matrixes probably contributed to their hydrolytic and biochemical stability, due to the hydrophobicity of this monomer. Yap and others[21] also showed that the surface roughness of a Bis-EMA–based composite was not affected by acidic beverages. Bis-EMA shows a decreased flexibility and increased hydrophobicity due to the elimination of the hydroxyl groups when compared with composites formulated with Bis-GMA.[22] Hence, the reduction in water uptake may be partially responsible for the chemical stability of composites that contain Bis-EMA.

Regarding RMGIC, an acid environment has been shown to have a severe effect on surface degradation.[18] The degradation by orange juice or Coca-Cola caused a significant increase in roughness surface for Ketac N100 and Vitremer (Figure 1; Table 3). The presence of HEMA, a highly hydrophilic monomer in the organic matrix from RMGICs, can increase the solubility of RMGICs.[23] Rogalewicz and others[24] observed HEMA, TEGDMA, and additive decomposition products eluted from a RMGIC after seven days of immersion in acidic media. It is possible that the loss of components from the two Vitremer and Ketac N100 matrixes (polyacrylate-inorganic and polymer-organic) leads to changes in surface roughness and hardness. Therefore, it could be speculated that the acidic environment corroded the RMGIC

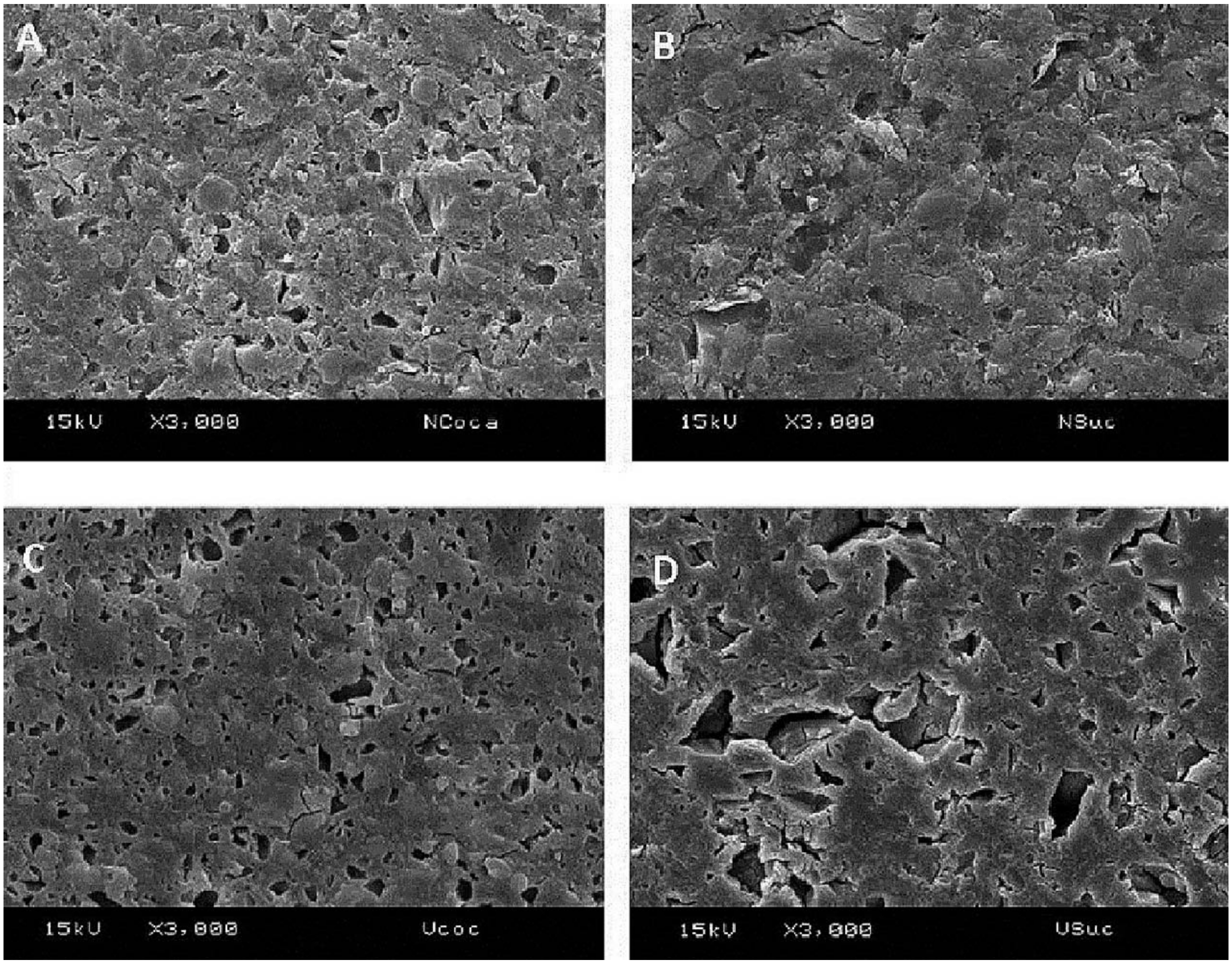

Figure 1. *Representative scanning electron micrographs of glass ionomer cement after degradation. (A): Ketac Nano/Coca-Cola. (B): Ketac Nano/ orange juice. (C): Vitremer/Coca-cola. (D): Vitremer/orange juice. Note corroded resin matrix provided by chemical erosion; extrusion fillers. Original magnification 3000×.*

matrix, promoting an increase in roughness (Figure 1; Table 3), also seen in micrographs. Thus, regarding roughness results, it is evident that the composition of the matrix influenced the surface roughness of materials subjected to a chemical challenge[25] and that the incorporation of nanoparticles in the composite and glass ionomer cement did not interfere with their chemical degradation resistance.

Hardness is a property that is used to predict the wear resistance of a material and was the parameter most affected by the chemical challenge in the current study. According to the present results, both composites (Filtek Z350 and TPH Spectrum) presented higher hardness values than the RMGICs. The different contents of organic matrixes and higher filler loading, as well as the higher degree of conversion for the resin composites, could explain the behavior of these materials compared with ionomers. However, their high filler loading (79% by weight, following manufacturer data) contributed to the similar initial hardness values between Filtek Z350 and TPH Spectrum, regardless of inclusion of nanofillers in only one.

After degradation in acidic beverages, all materials showed a significant reduction of hardness, and RMGICs showed a greater loss of hardness than the resin composites studied. The decreased hardness observed for all storage solutions seems to have originated from hydrolysis, since the more hydrophilic organic matrixes experienced greater hydrolysis.[26] According to Sakar,[27] corrosive wear begins with water absorption that diffuses internally through the resin matrix, filler interfaces, pores, and others defects, accelerated by the low pH of the solution. Thus, the chemical degradation rates of different materials depend on their hydrolytic stability, which is mainly related to the resin matrix.[28] As the resin matrix of composites is known

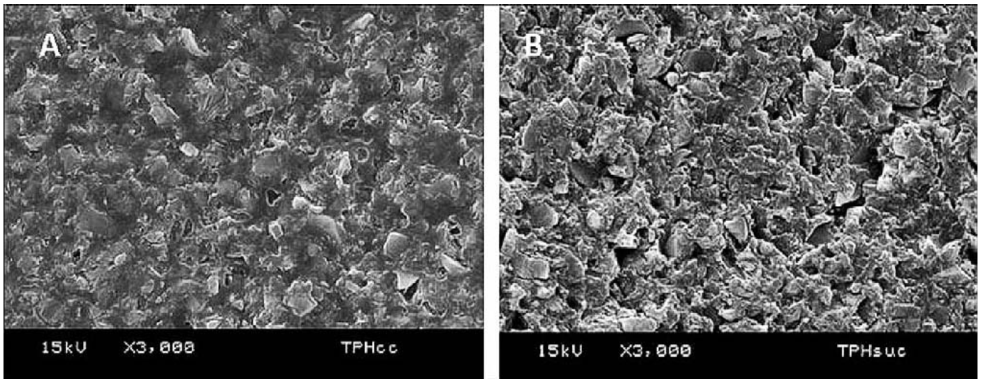

Figure 2. *Representative scanning electron micrographs of TPH Spectrum after storage in (A) Coca-Cola and (B) orange juice. Severe corroded aspect of the resin matrix pointed out by marking it after storage in orange juice (B). Original magnification 3000×.*

to absorb a small percentage of water,[29] composites were more resistant to degradation than were hydrophilic materials, such as RMGICs.[30] In addition, the storage solutions promoted dissolution near the glass particles, as seen in micrographs, which could be the result of dissolution of the siliceous hydrogel layer of RMGICs.[31]

It was observed that Filtek Z350 was the unique material that was not influenced by storage medium. This result can be supported by the hypothesis that the prime deleterious action resulted from the water and not from the acidic environment.[32] Despite a minor difference in the percentage load of the composites tested, the higher filler loading with smaller particle size provides a reduction in the interstitial spacing (less matrix exposition) and enhances the overall resistance of Filtek Z350 to chemical degradation[33] when compared with TPH Spectrum. Moreover, the greater part of TEGDMA from that resin composite was replaced with a blend of urethane dimethacrylate (UDMA) and Bis-EMA (ethoxylated bisphenol-A dimethacrylate). Pearson and Longman[34] determined that UDMA has lower water sorption than Bis-GMA, due to a higher conversion and cross-linking, evidencing the importance of the type of resin matrix in chemical degradation resistance.

Concerning the resin composite TPH Spectrum, a significant loss of surface hardness was observed after storage in orange juice. This could be related to its inorganic fillers, as suggested by Soderholm and others.[35] It was shown that materials containing barium glass fillers are more susceptible to acid attack. Moreover, the corrosive effect of the storage solutions did not depend only on their intrinsic pH value but also on their buffering effect, type, and chelating properties of the acid, among other acid characteristics.[36] According to Owens[37] and Cheng and others,[38] orange juice has a greater buffering capacity and corrosive effect than does Coca-Cola, explaining the lower hardness values of TPH Spectrum composite when stored in orange juice (Figure 2; Table 4). Still, the size and dimension of the citrate molecule induces the formation of stable complexes with metallic ions present at fillers.

Although all of the materials degraded with storage in all solutions, Coca-Cola and orange juice produced greater reductions in the hardness values for TPH Spectrum and RMGICs, with or without nanofiller inclusion.

CONCLUSION

It was concluded that different beverages (Coca-Cola and orange juice) provided great changes in surface roughness for RMGIC, regardless of the presence of nanofillers. Overall, Coca-Cola and orange juice provided a decrease in the hardness for all materials studied. The most intense decrease on hardness was observed for RMGICs immersed in both acidic solutions and TPH Spectrum in orange juice. Nanofillers did not influence the roughness and hardness of RMGIC or resin composites studied.

Conflict of Interest

The authors of this article certify that they have no proprietary, financial, or other personal interest of any nature or kind in any product, service, and/or company that is presented in this article.

(Accepted 15 July 2013)

REFERENCES

1. Fucio SB, Carvalho FG, Correr-Sobrinho L, Sinhoreti MAC, & Puppin-Rontani RM (2008) The influence of 30-day-old *Streptococcus mutans* biofilm on the surface of esthetic restorative materials: an in vitro study *Journal of Dentistry* **36(10)** 833-839.

2. Yap AUJ, Lim LY, Yang TY, Ali A, & Chung SM (2005) Influence of dietary solvents on strength of nanofill and ormocer composites. *Operative Dentistry* **30(1)** 129-133.
3. Badra VV, Faraoni JJ, Ramos RP, & Palma-Dibb RG (2005) Influence of different beverages on the microhardness and surface roughness of resin composites *Operative Dentistry* **30(2)** 213-219.
4. Finer Y, & Santerre JP (2004) The influence of resin chemistry on a dental composite‘s biodegradation *Journal of Biomedical Materials Research A* **69(2)** 233-246.
5. Fukazawa M, Matsuya S, & Yamane M (1990) The mechanism for erosion of glass-ionomer cements in organic-acid buffer solutions *Journal of Dental Research* **69(5)** 1175-1179.
6. Percq A, Dubois D, & Nicholson JW (2008) Water transport in resin-modified glass-ionomer dental cement *Journal of Biomaterials Application* **23(3)** 263-273.
7. Mitra SB, Wu D &, Holmes BN (2003) An application of nanotechnology in advanced dental materials *Journal of the American Dental Association* **134(10)** 1382-1390.
8. Senawongse P, & Pongprueksa P (2007) Surface roughness of nanofill and nanohybrid resin composites after polishing and brushing *Journal of Esthetic Restorative Dentistry* **19(5)** 265-273.
9. Taylor DF, Kalachandra S, Sankarapandian M, & McGrath JE (1998) Relationship between filler and matrix resin characteristics and the properties of uncured composite pastes. *Biomaterials* **19(1-3)** 197-204.
10. Beun S, Glorieux T, Devaux J, Vreven J, & Leloup G (2007) Characterization of nanofilled compared to universal and microfilled composites. *Dental Materials* **23(1)** 51-59.
11. Ketac Nano Light Curing Nano-Ionomer Restorative Technical product profile. Retrieved online October 10, 2010, from: http://www.3m.com/multimedia.mmm.com/mws/mediawebserver.dyn?6666660Zjcf6lVs6EVs66SJMhCOrrrrQ
12. de Fúcio SB, de Paula AB, de Carvalho FG, Feitosa VP, Ambrosano GM, & Puppin-Rontani RM (2012) Biomechanical degradation of the nano-filled resin-modified glass-ionomer surface. *American Journal of Dentistry* **25(6)** 315-320.
13. Carvalho F, Sampaio C, Fucio S, Carlo H, Correr-Sobrinho L, & Puppin-Rontani R (2012) Effect of chemical and mechanical degradation on surface roughness of three glass ionomers and a nanofilled resin composite. *Operative Dentistry* **37(5)** 509-517.
14. Topaloglu-Ak A, Cogulu D, Ersin NK, & Sen BH (2012) Microhardness and surface roughness of glass ionomer cements after APF and TiF4 applications. *Journal of Clinical Pediatric Dentistry* **37(1)** 45-51.
15. Bala O, Arisu HD, Yikilgan I, Arslan S, & Gullu A (2012) Evaluation of surface roughness and hardness of different glass ionomer cements. *European Journal of Dentistry* **6(1)** 79-86.
16. Perdigão J, Dutra-Corrêa M, Saraceni S, Ciaramicoli M, & Kiyan V (2012) Randomized clinical trial of two resin-modified glass ionomer materials: 1-year results. *Operative Dentistry* **37(6)** 591-601.
17. Aliping-McKenzie M, Linden RWA, & Nicholson JW (2004) The effect of Coca-Cola and fruit juices on the surface hardness of glass-ionomers and compomers *Journal of Oral Rehabilitation* **31(11)** 1046-1052.
18. Francisconi LF, Honório HM, Rios D, Magalhães AC, Machado MA, & Buzalaf MA (2008) Effect of erosive pH cycling on different restorative materials and on enamel restored with these materials *Operative Dentistry* **33(2)** 203-208.
19. Gladys S, Meerbeek BV, Braem M, Lambrechts P, & Vanherle G (1997) Comparative physico-mechanical characterization of new hybrid restorative materials with conventional glass-ionomer and resin composite restorative materials *Journal of Dental Research* **76(4)** 883-894.
20. Cavalcante LM, Masouras K, Watts DC, Pimenta LA, & Silikas N (2009) Effect of nanofillers’ size on surface properties after toothbrush abrasion. *American Journal of Dentistry* **22(1)** 60-64.
21. Yap AU, Low JS, & Ong LF (200) Effect of food-simulating liquids on surface characteristics of composite and polyacid-modified composite restoratives *Operative Dentistry* **25(3)** 170-176.
22. Wu W, & McKinney JE (1982) Influence of chemicals on wear of dental composites *Journal of Dental Research* **61(10)** 1180-1183.
23. Bagheri R, Burrow MF, & Tyas M (2005) Influence of food-simulating solutions and surface finish on susceptibility to staining of aesthetic restorative materials *Journal of Dentistry* **33(5)** 389-398.
24. Rogalewicz R, Batko K, & Voelkel A (2006) Identification of organic extractables from commercial resin-modified glass-ionomers using HPLC-MS. *Journal Environmental Monitoring* **8(7)** 750-758.
25. Loomans BA, Cardoso MV, Opdam NJ, Roeters FJ, De Munck J, Huysmans MC, & Van Meerbeek B (2011) Surface roughness of etched composite resin in light of composite repair *Journal of Dentistry* **39(7)** 499-505.
26. Prakki A, Cilli R, Mondelli RF, Kalachandra S, & Pereira JC (2005) Influence of pH environment on polymer based dental materials properties. *Journal of Dentistry* **33(2)** 91-98.
27. Sarkar NK (2000) Internal corrosion in dental composite wear: its significance and simulation. *Journal of Biomedical Materials Research* **53(4)** 371-380.
28. Asmussen E (1984) Softening of Bis-GMA-based polymers by ethanol and by organic acids of plaque *Scandnavica Journal of Dental Research* **92(3)** 257-261.
29. Ferracane JL (2006) Hygroscopic and hydrolytic effects in dental polymer networks *Dental Materials* **22(3)** 211-222.
30. Mohan M, Shey Z, Vaidyanathan I, Vaidyanathan TK, & Sivakumar M (2008) Color changes of restorative materials exposed in vitro to cola beverage. *Pediatric Dentistry* **30(4)** 309-316.
31. Turssi CP, Hara AT, Serra MC, & Rodrigues AL Jr (2002) Effect of storage media upon the surface micromorphology of resin-based restorative materials. *Journal of Oral Rehabilitation* **29(9)** 864-871.

32. Honório HM, Rios D, Francisconi LF, Magalhães AC, Machado MA, & Buzalaf MA (2008) Effect of prolonged erosive pH cycling on different restorative materials *Journal of Oral Rehabilitation* **35(12)** 947-953.

33. Wongkhantee S, Patanapiradej V, Maneenut C, & Tantbirojn D (2006) Effect of acidic food and drinks on surface hardness of enamel, dentine, and tooth-coloured filling materials *Journal of Dentistry* **34(3)** 214-220.

34. Pearson GJ, & Longman CM (1989) Water sorption and solubility of resin-based materials following inadequate polymerization by a visible-light curing system *Journal of Oral Rehabilitation* **16(1)** 57-61.

35. Soderholm KG, Zigan M, Ragan M, Fischlschweiger W, & Bergman M (1984) Hydrolytic degradation of dental composites *Journal of Dental Research* **63(10)** 1248-1254.

36. Lussi A, Jaeggi T, & Jaeggi-Scharer S (1995) Prediction of the erosive potential of some beverages *Caries Research* **29(5)** 349-354.

37. Owens BM (2007) The potential effects of pH and buffering capacity on dental erosion *General Dentistry* **55(6)** 527-531.

38. Cheng R, Yang H, Shao M, & Hu T (2009) Dental erosion and severe tooth decay related to soft drinks: a case report and literature review *Journal of Zhejiang University Science B* **10(5)** 395-339.