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Comparative Evaluation of Microleakage Among Three Different Glass Ionomer Types

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Clinical Relevance

Microleakage was evident in all restorative materials. Improvement was made when a resin-modified glass ionomer incorporated the benefits of nanotechnology.

ABSTRACT

Objectives: This study compared the microleakage and adaptation of Class V cavity preparations restored with three types of glass-ionomer materials as a function of time. Methods and Materials: A total of 144 sound, freshly-extracted human premolars were used for the study. One clinician prepared all the teeth for Class V-type cavities on the buccal surface of each tooth. The preparations measured 3 mm long, 2 mm wide and 1.5 mm deep, with the gingival margin in dentin and the occlusal margin in enamel. All the prepared teeth were randomly divided into three groups of 48 teeth, according to the type of glass-ionomer material used: Group (A): Ketac N100 glass ionomer, Group (B): Vitremer glass ionomer and Group (C): Photac Fil Quick glass ionomer. The restorative materials were used according to their manufacturers' recommendations. The teeth were placed in one increment and photocured for 40 seconds. All of the restored teeth were then stored in artificial saliva. Each group was subdivided into three subgroups according to the testing periods (7, 30, 60 days). Next, they were thermocycled at 5°C-55°C for 100 cycles. The teeth used for the dye penetration test were immersed in 1% methylene blue solution for eight hours. They were then sectioned longitudinally in a bucco-lingual direction. The extent of dye penetration at the occlusal and gingival margins of each restoration was studied under a stereomicroscope at 25x magnification. Randomly selected samples from each group were prepared for scanning electron microscopic evaluation. Dye penetration scores were analyzed using the SAS program, cross tabulation and Chi square test. Results: The difference among the three

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groups was significant after immersion for 30 days at the occlusal margin. Statistical analysis also revealed significant differences between group (A) and the other groups at the occlusal margin after immersion for 60 days (p<0.05). At the gingival margin, statistical analysis revealed significant differences between group (C) and the other groups at the gingival margin after immersion for 60 days (p<0.05).

Conclusion: The light-curing nanofilled glass ionomer (Ketac N100) showed the least microleakage.

INTRODUCTION

There has always been a keen interest in the adaptation of dental restorative materials to the walls of cavities and the retentive ability of a material to seal the cavity against the ingress of oral fluids and microorganisms. Microleakage around dental restorative materials is a major problem in clinical dentistry. It may be defined as the clinically undetectable passage of bacteria, fluids, molecules or ions between a cavity wall and the restorative materials applied to it.[1] This seepage can cause hypersensitivity of the restored teeth, tooth discoloration, recurrent caries, pulpal injury and accelerated deterioration of some restorative materials. Most restorative materials show varying degrees of marginal leakage because of dimensional changes and a lack of adaptability to cavity walls.[2]

Over the past 50 years, many changes have occurred in the development and availability of restorative materials. Glass ionomer cements (GICs) are adhesive bio-active restorative materials with therapeutic action that were developed during the late 1960s. GICs are advantageous for use in restorative dentistry due to their capacity to bond to dentin, fluoride release that helps remineralization, and biocompatibility. Due to the match in coefficient of thermal expansion between the tooth structure and GICs, they provide good marginal sealing, little microleakage and a high retention rate. Despite these advantages, conventional GICs have a number of clinical limitations, including prolonged setting time, dehydration during initial setting and a rough surface texture that can hamper mechanical resistance.[3] To overcome these shortcomings, a light-cured resin-modified glass ionomer (RMGIC) was introduced in the early 1990s, which contains polymerizable monomers and a photoinitiator in additional polyacrylic acid. Compared with conventional analogs, RMGICs have a longer working time, a rapid set, improved appearance and translucency and higher early strength.[4]

Recently, a light-curing Nano-Ionomer restorative was the first resin-modified glass-ionomer material developed with nanotechnology. It may add benefits not usually associated with glass ionomers because the filler particle size can influence strength, optical properties and abrasion resistance.[5-6]

The majority of cervical lesions exhibit mixed cavity margins positioned in both enamel and dentin.[7] Enamel possesses an important quantity of hydroxyapatite and its organic tissue is in smaller proportions. On the other hand, dentin is made of two different substrates: intertubular dentin, which is less mineralized, and peritubular dentin, which is more mineralized. The presence of water in dentin decreases surface energy and prevents restorations from establishing good mechanical retention.[7]

The current study evaluated microleakage and the adaptation of Class V cavity preparations restored with a nanofilled glass ionomer, a resin-modified glass ionomer and a modified synthetic glass polyalkenoate.

METHODS AND MATERIALS

A total of 144 sound, freshly obtained human premolars extracted for periodontal reasons were selected and used for the current study. Residual soft tissue was removed by scaling and storing the teeth in distilled water containing thymol crystals during the interval between extraction and their use in this *in vitro* study. One clinician prepared all the teeth for Class V-type cavities on the buccal surface of each tooth using an 835-010-4 ML cylindrical diamond bur (Diatech Dental, Coltène Whaldent AG, Alstätten, Switzerland) under air-water cooling. The bur was replaced every four preparations. The preparations measured 3 mm long, 2 mm wide and 1.5 mm deep, with the gingival margin in dentin and the occlusal margin in enamel. All the prepared teeth were randomly divided into three groups of 48 teeth according to the type of glass-ionomer material used, as follows:

Group (A): Ketac N100 (3M ESPE, St Paul, MN, USA); Group (B): Vitremer (3M ESPE) and Group (C): Photac Fil Quick (3M ESPE). The commercial name, composition and manufacturer of the three materials used in this study are listed in Table 1.

The restorative materials were used according to their manufacturer's recommendations. The restorative materials were placed in one increment, since the depths were less than 2 mm. They were photocured for 40 seconds using a visible Quartz-Tungsten-Halogen light-curing unit (Cromalux 7050 [Mega-PHYSIK GmbH & Co KG, Megadenta, Germany] at 500mW/cm^2). The restorations were finished immediately with fine-grit finishing diamond burs and polished with a graded series of flexible discs (Sof-Lex, 3M ESPE). All the restored teeth were stored in artificial saliva (0.4g NaCl–0.4g KCl–0.795g $CaCl_2.2H_2O$–0.69g Na_2HPO_4–0.005g Na_2S. $9H_2O$–1g Urea + 1 L Deionized water) pH (7.03) at body temperature.[8] Each group was

Table 1: *The Commercial Name, Composition and Manufacturer of the Materials Used*

Materials	Manufacturer	Composition
Ketac N100 Light-Curing Nano-Ionomer Restorative	3M ESPE, St Paul, MN, USA	Deionized water, blend, including HEMA, a methacrylate-modified polyalkenoic acid. Filler component: methacrylate functional-fluoroaluminosilicate glass and nanomeres and nano-clusters.
Vitremer	3M ESPE, St Paul, MN, USA	The powder is a radiopaque, fluoroaluminosilicate glass. The liquid is a light sensitive, aqueous solution of a modified polyalkenoic acid.
Photac Fil Quick	3M ESPE, St Paul, MN, USA	The powder contains highly polishable, radiopaque glass particles (Na-Ca-Al-Lafluorosilicate-glass) and amines as activators for light-curing. The liquid component contains copolymer acids (maleic and acrylic acid), camphorquinone as an initiator of the light-curing and selected glass ionomer compatible monomers and oligomers.

then divided into three subgroups according to the testing periods (7, 30, 60 days). They were then thermocycled at 5°C-55°C for 100 cycles with a 20-second dwell time at each temperature and a 10-second transfer time, for a total of 50 seconds per cycle.

Preparation of the Teeth for Dye Penetration Test

The apices of the roots were sealed with modeling wax and the whole tooth was covered with two layers of nail polish, leaving a 1 mm window all around the cavity margin. The teeth were immersed in a 1% aqueous solution of methylene blue for eight hours, then removed from the dye, brushed thoroughly under tap water for 30 seconds and the wax was removed with a wax knife.

The teeth were sectioned longitudinally in a bucco-lingual direction through the center of the restorations using a water-cooled low-speed diamond saw (Isomet, Buehler, Ltd, Lake Bluff, IL, USA).

The section with the greater leakage was evaluated with a stereomicroscope (M9, Wild Heerbrugg, Switzerland) at 25x magnification by a single operator who was blinded as to which group each sample came from to determine the extent of dye penetration at the occlusal and gingival margins. Scoring was done according to the following criteria suggested by Koenigsberg and others:[9]

0- No dye penetration

1- Dye penetration in enamel to the dentino-enamel junction (DEJ)

2- Dye penetration in dentin midway between the distance of the DEJ to the axial wall

3- Dye penetration up to the axial wall

SEM Analysis

Four randomly selected samples from each group were prepared for scanning electron microscopic evaluation. The specimens were dehydrated and mounted on aluminum stubs, then gold-sputter coated. The SEM (JEOL JXA-840A Electron probe Micro-analyzer SEM, JOEL, Ltd, Tokyo, Japan) was then used to assess marginal adaptation of the three tested materials to tooth structure at 1500x magnification.

Statistical Analysis

The data were analyzed using the SAS program (SAS, 1988). Cross-tabulation and the Chi-square test were used to test the effects of the material within each time and the effect of time within each material on the dye penetration score.

RESULTS

Figures 1 and 2 show the percentage of leakage scores at the occlusal and gingival margins for the three types of glass ionomer that were examined after immersion for three different time periods and thermocycling.

Comparison of the dye penetration scores of the three glass-ionomer types (groups A, B and C) revealed no

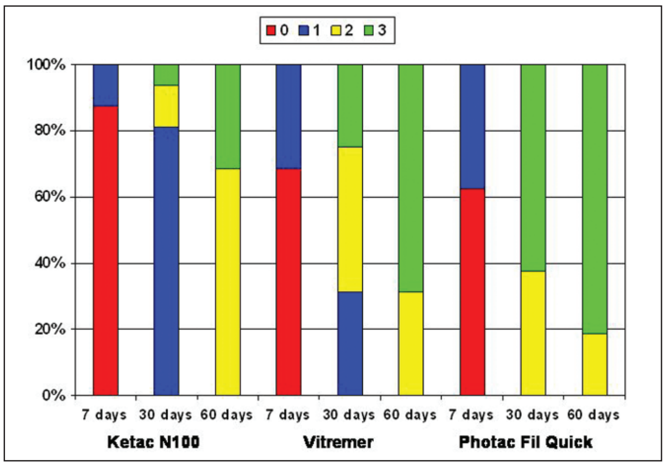

Figure 1. *Prevalence of dye penetration scores of the three tested glass-ionomer types during the follow-up period (occlusal).*

significant differences at the occlusal margin or the gingival margin after immersion for seven days ($p<0.05$). However, the difference was significant among the three groups after immersion for 30 days at the occlusal margin. Statistical analysis also revealed insignificant differences between groups B and C, while the difference was significant between these two groups and group A at the occlusal margin after immersion for 60 days ($p<0.05$).

At the gingival margin, statistical analysis revealed no significant difference between groups A and B, while the difference was significant between these two groups and group C after immersion for 30 days. Statistical analysis also revealed insignificant differences between groups A and B, while the difference was significant between these two groups and group C at the gingival margin after immersion for 60 days ($p<0.05$).

Regarding Ketac N100 (3M ESPE) and Vitremer (3M ESPE) glass-ionomer types, the difference was significant between specimens immersed for 7 days, 30 days and 60 days ($p<0.05$) at both the occlusal and gingival margins, where leakage increased by increasing the immersion time. On the other hand, Photac-Fil Quick (3M ESPE) revealed a significant difference between specimens immersed for seven days and those immersed for 30 and 60 days, where no significant difference resulted between them at the occlusal margins. The difference was significant after the immersion of Photac-Fil Quick for 7 days, 30 days and 60 days at the gingival margin.

Figures 1 and 2 show that Ketac N100 (3M ESPE) allowed the least leakage at both margins, followed by Vitremer, then Photac-Fil Quick.

Finally, statistical analysis comparing the prevalence of dye penetration scores at the occlusal versus the gingival margin within each material and immersion time revealed a significant difference between

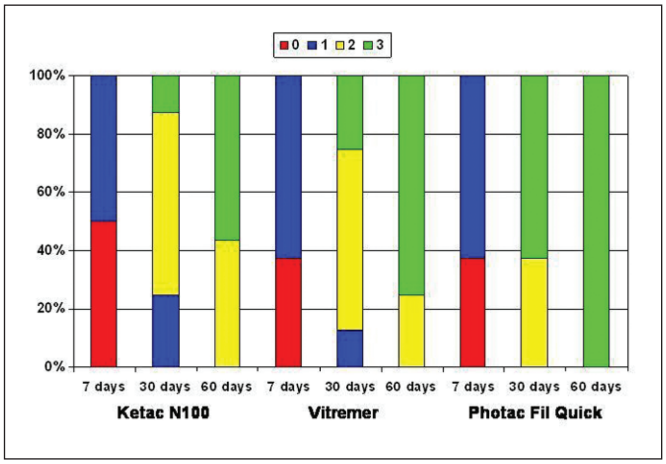

Figure 2. *Prevalence of dye penetration scores of the three tested glass-ionomer types during the follow-up period (gingival).*

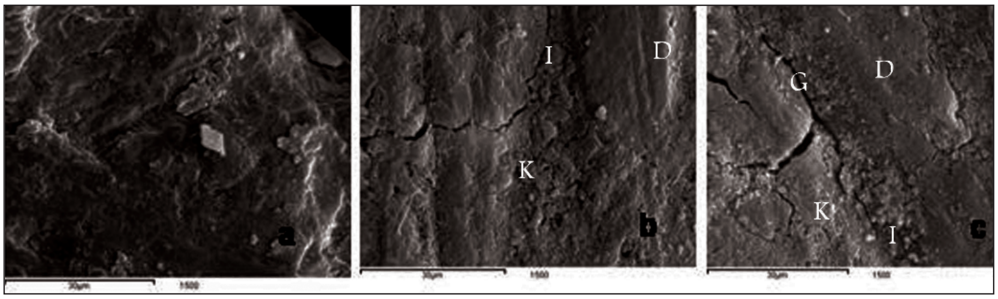

Figure 3. *High resolution SEM micrograph of marginal adaptation of Ketac N100 glass ionomer type after immersion for Figure 3a: 7 days; Figure 3b: 30 days and Figure 3c: 60 days. (K) Ketac N100. (I) K/dentin interface. (D) Dentin. (G) Gap (1500x).*

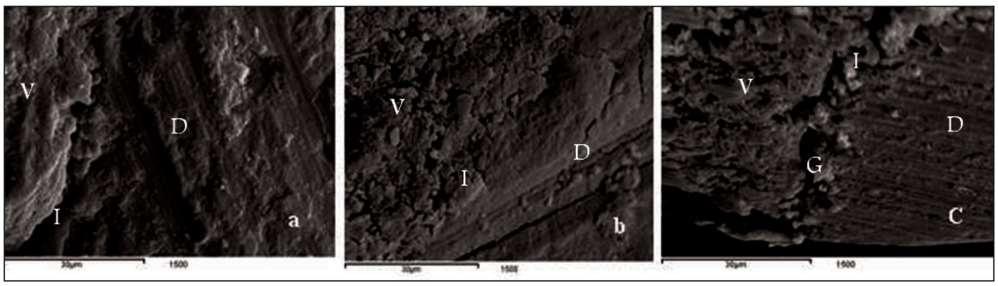

Figure 4. *High resolution SEM micrograph of marginal adaptation of Vitremer glass ionomer type after immersion for Figure 4a: 7 days; Figure 4b: 30 days and Figure 4c: 60 days. (V) Vitremer. (I) V/dentin interface. (D) Dentin. (G) Gap (1500x).*

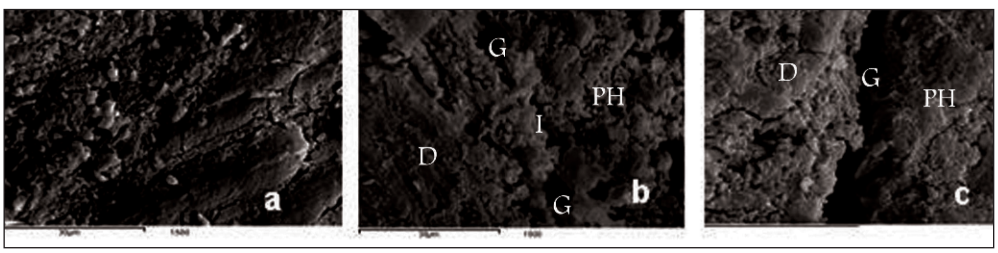

Figure 5. *High resolution SEM micrograph of marginal adaptation of Photac Fil Quick glass ionomer type after immersion for Figure 5a: 7 days; Figure 5b: 30 days and Figure 5c: 60 days. (PH) Photac Fil Quick. (I) PH/dentin interface. (D) Dentin. (G) Gap (1500x).*

the occlusal and gingival margin in group A after immersion for 7 and 30 days, while the difference was insignificant between the two margins after immersion for 60 days. On the other hand, there was insignificant difference between the two margins in groups B and C at all the tested immersion time periods ($p<0.05$).

SEM Results

Gaps between the tooth structure and cements were less in the Ketac N100 glass-ionomer type (Figure 3) than gaps found in the other two tested glass-ionomer types (Figures 4 and 5), regardless of the immersion period. This finding was inconsistent with the dye penetration values achieved. More gaps appeared in the Photac Fil Quick glass-ionomer type immersed for 60 days (Figure 5c) than the other tested material types immersed for the same period (Figures 3c and 4c). In the three tested types of materials, the gaps were more obvious when increasing the immersion period. Higher magnification of the bond interface of Ketac N100 specimens stored for seven days showed an indistinct interface between the margin of the tooth structure and the restoration, suggesting that a chemical bond had formed between the GIC and tooth structure (Figure 3a). It was also difficult to distinguish between the tooth structure and Ketac N100 GIC-type due to its nano microstructure.

DISCUSSION

The current study examined the microleakage of different types of glass-ionomer restorations placed in Class V cavities using a dye penetration test. Microleakage is an important property that has been used in assessing the success of any restorative material used in tooth restorations.[2-10]

Thermocycling is a standard protocol in the restorative literature when bonded materials are evaluated, simulating *in vivo* aging by subjecting bonded materials to cyclic exposures of hot and cold temperatures.[11] A novel RMGI restorative filling material, Ketac N100 (3M ESPE), has been developed, incorporating the benefits of nanotechnology.[12]

As regards the occlusal margin, the results of the current study showed no significant difference among the three tested types of glass ionomer after immersion for seven days. On the other hand, the difference was significant among the three tested groups after immersion for 30 days. Moreover, there was a significant difference between Ketac N100 and the other two tested glass-ionomer types after immersion for 60 days. The nanofilled type of glass ionomer (Ketac N100) showed the lowest microleakage scores, followed by the resin-modified glass ionomer (Vitremer), then the modified synthetic glass polyalkenoate (Photac Fil Quick). These types of glass ionomers are resin-modified glass ionomers, where two types of setting reactions occur: 1) the acid-base reaction between the fluoroaluminosilicate glass and the polycarboxylic acid and 2) a light-activated free radical polymerization of the methacrylate groups of the polymer and HEMA (2-hydroxyethylmethacrylate).[13-15] Moreover, the actual bonding mechanism of RMGIs to tooth tissues has recently been determined to be twofold by micromechanical interlocking [16-17] and chemical interaction.[18-19]

The basic bonding mechanism was an ionic attraction between two carboxyl (COO-) groups in the cement to calcium (Ca++) in enamel and dentin.[20] Micromechanical interlocking is achieved by impregnation of a partially-demineralized layer on the top of the dentin substrate, with a high-molecular-weight polycarboxyl-based polymer.[21] Additional bonding mechanisms have been explored for the RM-GICs, since the presence of resin suggests that bonding analogous to resin composite may occur; that is, resin tags into enamel and the establishment of a hybrid layer from the hydrophilic HEMA.[22]

Light-activated polymerization is accompanied by a certain degree of polymerization shrinkage that takes place in the three tested materials.[6-23] Vitremer and Photac Fil Quick contain fluoroaluminosilicate glass, while the nanofilled glass-ionomer type contains fluoroaluminosilicate glass, together with nanomers and nanoclusters in the filler loading, which is approximately 69% by weight. The higher filler loading in the nanofilled type may result in lower polymerization shrinkage and lower coefficient of thermal expansion of this type of glass ionomer, improving the long-term bonding to tooth structure. An *in vitro* study has shown that a controversy exists as to whether slight polymerization shrinkage is significant enough to disrupt the margin seal.[24]

The higher leakage produced by the Photac Fil Quick type may be due to the fact that no primer was used with this type of glass ionomer, while the other types get the benefit of using primer that is acidic in nature. Its function is to modify the smear layer and adequately wet the tooth surface to facilitate adhesion of the material to the hard tissue; this is in agreement with other studies that concluded conditioning plays a greater role in achieving effective bonding with resin-modified glass-ionomer cements.[25] The Photac Fil Quick type showed initial low leakage scores, but the leakage scores increased by increasing storage time, indicating unstable bonding to tooth structure.

With all of the tested materials, the results showed higher leakage scores at the gingival margin than the occlusal margin and at different tested immersion times. This finding is in agreement with other studies that concluded, while cavity preparations with enamel margins result in consistently stronger bonds, unique challenges are encountered with dentin surface bonding due to enamel that is 92% inorganic hydroxyapatite and dentin that is 45% inorganic by volume.[26-27] Statistical analysis showed a statistically significant difference in leakage between the occlusal and gingival margins with Ketac N100 after immersion for 7 and 30 days, while the difference was insignificant after immersion for 60 days. This may be explained by the fact that the nanostructure of the nanofilled glass-ionomer type allowed for excellent wetting and adaptability to the tooth surface, hence enhancing the chemical bonding. This fact was inconsistent with the scanning electron microscope results, where it was difficult to distinguish between the nanofilled glass ionomer and tooth structure. Since chemical bonding takes place through a chelation reaction with calcium on the surface of the tooth, its effect was more significant on the occlusal side with an enamel margin than the gingival side with only a dentin margin. The difference was insignificant between the occlusal and gingival margins after immersion for 60 days, indicating an unstable bonding where leakage eventually increased at the occlusal margin.

CONCLUSIONS

Within the limitations of the current study, it could be concluded that:

1- Leakage will eventually occur with all types of glass ionomer with an increase in immersion time.

2- Photac Fil Quick glass ionomer showed the most leakage, followed by Vitremer glass ionomer. The light-curing nanofilled type of glass ionomer (Ketac N100) showed the least microleakage.

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References

1. Morabito A & Defabianis P (1997) Marginal seal of various restorative materials in primary molars *Journal of Clinical Pediatric Dentistry* **22(1)** 51-54.
2. Mali P, Deshpande S & Singh A (2006) Microleakage of restorative materials: An *in vitro* study *Journal of Indian Society of Pedodontics and Preventive Dentistry* **24(1)** 15-18.
3. Bona A D, Pinzetta C & Rosa V (2009) Microleakage of acid etched glass ionomer sandwich restorations *Journal of Minimum Intervention in Dentistry* **2(1)** 36-43.
4. Katleen I M, Peter J & Mieke AA (2008) Microleakage of four different restorative glass ionomer formulations in Class V cavities: Er:YAG laser versus conventional preparation *Photomedicine and Laser Surgery* **26(6)** 541-549.
5. Xie D, Brantley WA & Culbertson BM (2000) Mechanical properties and microstructures of glass ionomer cements *Dental Materials* **16(2)** 129-138.
6. Wadenya R, Smith J & Mante F (2010) Microleakage of nano-particle-filled resin-modified glass ionomer using atraumatic restorative technique in primary molars *New York State Dental Journal* **76(4)** 36-39.
7. Santini A, Plasschaert A & Mitchell S (2000) Marginal leakage of filled dentin adhesives used with wet and dry bonding techniques *American Journal of Dentistry* **13(2)** 93-97.
8. Robin A & Meirelis JP (2007) Influence of fluoride concentration and pH on corrosion behavior of titanium in artificial saliva *Journal of Applied Electrochemistry* **37(4)** 511-517.
9. Koenigsberg S, Fuk A & Grajower R (1989) The effect of three filling techniques on marginal leakage around Class II composite resin restorations *in vitro Quintessence International* **20(2)** 117-121.
10. Hollan JR, Nystrom GF, Douglas WH & Phelps RA (1990) Microleakage and marginal placement of a glass-ionomer liner *Quintessence International* **21(2)** 117-122.
11. Nalcaci A & Ulusoy N (2007) Effect of thermocycling on microleakage of resin composites polymerized with LED curing techniques *Quintessence International* **38(616)** 433-439.
12. Croll TP (2007) Nanofilled resin-modified glass ionomer restorative cement *Contemporary Esthetics* **11(9)** 14-17.
13 Quo BC, Drummond JL & Koerber A (2002) Glass ionomer microleakage from preparations by an Er/YAG laser or a high-speed handpiece *Journal of Dentistry* **30(4)** 141-146.
14. Rossi RR, Aranha AC, Eduardo CP, Ferreira LS, Navarro RS & Zezell DM (2008) Microleakage of glass ionomer restoration in cavities prepared by Er,Cr:YSGG laser irradiation in primary teeth *Journal of Dentistry for Children* **75(2)** 151-157.
15. Berzins DW, Abey S, Costache MC, Wilkie CA & Roberts HW (2010) Resin-modified glass-ionomer setting reaction competition *Journal of Dental Research* **89(1)** 82-86.
16. Tay FR, Smales RJ, Ngo H, Wei SH & Pashley DH (2001) Effect of different conditioning protocols on adhesion of a GIC to dentin *Journal of Adhesive Dentistry* **3(2)** 153-167.
17. Yip HK, Tay FR, Ngo HC, Smales RJ & Pashley DH (2001) Bonding of contemporary glass ionomer cements to dentin *Dental Materials* **17(5)** 456-470.
18. Yoshida Y, Van Meerbeek B, Nakayama Y, Snauwaert J, Hellemans L & Lambrechts P (2000) Evidence of chemical bonding at biomaterial-hard tissue interfaces *Journal of Dental Research* **79(2)** 709-714.
19. Fukuda R, Yoshida Y, Nakayama Y, Okazaki M, Inoue S & Sano H (2003) Bonding efficacy of polyalkenoic acids to hydroxyapatite, enamel and dentin *Biomaterials* **24(11)** 1861-1867.
20. Tyas MJ & Burrow MF (2004) Adhesive restorative materials: A review *Australian Dental Journal* **49(3)** 112-121.
21. Van Meerbeek B, De Munck J, Yoshida Y, Inoue S, Vargas M & Vijay P (2003) Buonocore Memorial Lecture. Adhesion to enamel and dentin: Current status and future challenges *Operative Dentistry* **28(3)** 215-235.
22. Coutinho E, Van Landuyt K, De Munck J, Poitevin A, Yoshida Y, Inoue S, Peumans M, Suzuki K, Lambrechts P & Van Meerbeek B (2006) Development of a self-etch adhesive for resin-modified glass ionomers *Journal of Dental Research* **85(4)** 349-353.
23. Chong BS, Ford TR & Watson TF (1991) The adaptation and sealing ability of light-cured glass ionomer retrograde root fillings *International Endodontic Journal* **24(5)** 223-232.

24. Sidhu SK & Watson TF (1995) Resin-modified glass ionomer materials. A status report for the American Journal of Dentistry *American Journal of Dentistry* **8(1)** 59-67.

25. Cho S & Cheng AC (1999) A review of glass ionomer restorations in the primary dentition *Journal of the Canadian Dental Association* **65(9)** 491-495.

26 Phair CB & Fuller JL (1985) Microleakage of composite resin restorations with cementum margins *Journal of Prosthetic Dentistry* **53(3)** 361-364.

27. Swift E, Perdigão J & Heymann HO (1995) Bonding to enamel and dentin: A brief history and state of the art *Quintessence International* **26(2)** 95-110.