J Mater Sci: Mater Med (2007) 18:1465–1470
DOI 10.1007/s10856-006-0084-z

Analysis of surfaces and adhesive interfaces of enamel and dentin after different treatments

Michelle Alexandra Chinelatti · Silmara Aparecida Milori Corona · Maria Cristina Borsatto · Lilian Faria Ribeiro · Renata Andréa Salvitti de Sá Rocha · Regina Guenka Palma-Dibb

Received: 29 August 2005 / Accepted: 22 February 2006 / Published online: 27 March 2007

Abstract
Purpose The purpose of this study was to analyze, by Scanning Electron Microscopy (SEM), the surface topography and the morphology of the adhesive interfaces of enamel and dentin after different treatments.
Materials and methods The enamel–dentin discs were randomly assigned into three groups according to the surface treatment: I—37% phosphoric acid; II—air-abrasion; III—air-abrasion followed by 37% phosphoric acid. After surface treatment, discs were divided in two: one hemi-disc was separated for surface analysis; the other hemi-disc received the Single Bond/Filtek Z-250 restorative system. The restored sections were bisected perpendicularly to the surface and prepared for interface analysis.
Results Results disclosed that when the surface treatment was performed by air-abrasion, irregularities were observed at the enamel surface; microcracks and occluded tubules at dentin surface and lack of hybrid layer at adhesive/dentin interface. The air-abrasion treatment followed by acid etching provided an enamel etching pattern similar to the acid etching; microfissures and open tubules at dentin surface, and formation of hybrid layer at adhesive-dentin interface.
Conclusion It may be concluded that the treatment with air-abrasion followed by acid etching is an effective procedure to obtain an adequate surface for resin adhesion.

M. A. Chinelatti (✉) · S. A. M. Corona ·
L. F. Ribeiro · R. G. Palma-Dibb
Department of Restorative Dentistry, Ribeirão Preto School of Dentistry, College of Odontologia of Ribeirão Preto, University of São Paulo, Av. do Cafe, S/N, 14040-904 Ribeirao Preto, SP, Brazil
e-mail: michinelatti@gmail.com

M. C. Borsatto · R. A. Salvitti de Sá Rocha
Department of Pediatric Dentistry, Ribeirão Preto School of Dentistry, University of São Paulo, Ribeirao Preto, Brazil

Introduction

Initially introduced by Buonocore [1] in 1955, acid-etch bonding technique has significantly changed the clinical practice in all fields of dentistry. Modern direct esthetic dentistry is based in large part on adhesive bonding of composite resins to enamel and dentin. The bond to the tooth is mediated by an adhesive system, which is applied after conditioning enamel and dentin with the acid-etching technique (total etch) [2], or by the self-etching adhesives, which is based on the use of polymerizable acidic monomers that simultaneously condition and prime dentin and enamel [3]. The purpose of the low-viscosity adhesive systems is to penetrate into the decalcified tooth structure and form a micromechanical retention by formation of resin tags into the enamel etch pattern and open dentin tubules and to hybridize the collagenous network of the dentin [2–6].

Air-abrasion technique has been described as another method of tooth structure pretreatment. The first air-abrasion unit was introduced by Black [7] in 1945 for nonmechanical cavity preparation and prophylaxis. Air-abrasion technology uses a high-speed stream of aluminum oxide particles propelled by air-pressure, which, when they impact on the tooth surface, results in an effective and rapid substance removal [8–10].

Several researches involving air-abrasion for cavity preparations [10–14], enamel surface treatment [15–20], dentin surface treatment [21–23] and caries removal [24–29] have been carried out in order to preserve healthy dental tissues and increase the bonding characteristics of restorative materials, thereby providing a more comfortable and less stressing procedure for the patient [30–34]. Air-abrasion provides a rough irregular surface with large surface area and increases its wettability for the adhesive system [22, 35, 36]. In order to better understand the mechanisms of adhesion of restorative materials, several studies [10, 19, 20, 22, 23] were conducted assessing the morphological structure, by SEM, of enamel and dentin surfaces after air-abrasion application. Some authors [10, 22] have reported the presence of aluminum oxide particles on the air-abraded surface and fissures and occluded tubules at dentin, differing thereby from the pattern provided by acid-etch.

However, there are divergent results in the current literature concerning the influence of air-abrasion on tooth surface morphology and adhesive interface between composites and enamel or dentin. Based on this, the conducted study aimed to analyze, employing Scanning Electron Microscopy, the morphology of adhesive-enamel and adhesive-dentin interfaces as well as enamel and dentin surfaces resulting from different treatments.

Materials and methods

Sound human molars, extracted within a 6-month period, were cleaned with scaler and water/pumice slurry in dental prophylactic cups and examined under a ×20 magnifier to discard those with structural defects. Twelve molars were selected for the study and stored in distilled water at 4° C.

Teeth were taken to the sectioning machine and the occlusal overlying enamel surface was eliminated with the water-cooled diamond saw at low-speed to prevent fracture or overheating. Then, for each tooth, another cut was accomplished in a mesio-distal direction, providing a 1.0 mm-thick disc of dentin with enamel margins, which was bisected in a bucco-lingual direction, thus resulting in two hemi-discs. The hemi-discs had their pulpal surface isolated with two layers of a colorless nail varnish to prevent the release of organic solvents of the adhesive system through dentine tubules. The dentin surface corresponding to the occlusal side of the hemi-discs was polished with #600-grit SIC papers for 30 s to produce a standardized smear layer. The hemi-discs were randomly assigned to three groups ($n = 8$), according to the surface treatment:

Group I: enamel and dentin surfaces were etched with 37% phosphoric acid gel (Scotchbond etchant, 3M/ESPE, St. Paul, MN, USA) for 15 s, rinsed thoroughly for 15 s and gently dried with absorbing paper to remove the excess of water and keep the surface moist.

Group II: enamel and dentin surfaces were treated with an air-abrasion system (Mach 4.1, Kreativ Inc., Albany, OR, USA), with 27.5 μm-aluminum oxide particles, under 60 psi pressure and intensity of 4 g/min at continuous mode, delivered by a 0.011-inch nozzle opening, under a 45° angle with the surface of the disc. The application distance was standardized using a custom designed apparatus consisting of a moving holder that positioned the handpiece in such way that the aluminum oxide particles stream was delivered at a constant distance of 2 mm from the surface of the disc. The specimens were fixed with wax at a semi-adjustable base. The operator manipulated in such way that the semi-adjustable base with the disc was moved in right-to-left and forward-to-back directions, thereby allowing the stream to provide a more accurate application of the entire surface. The length of application was 10 s.

Group III: enamel and dentin surfaces were treated with air-abrasion and subsequent application of 37% phosphoric acid gel, following the same experimental conditions as described for Groups I and II.

Then, four hemi-discs of each Group were separated for surface analysis. The other half were subjected to the restorative procedure: two consecutive layers of a single-bottle adhesive (Single Bond, 3M/ESPE, St. Paul, MN, USA) were applied, the remaining solvent was evaporated with a brief, mild air-blast and the adhesive was light-cured for 10 s with a visible light curing unit (XL 3000, 3M/ESPE, St. Paul, MN, USA) with a 450 mW/cm^2 output. A hybrid light-curing composite resin (Filtek Z-250, 3M/ESPE, St. Paul, MN, USA) was inserted in three increments of 1.0–2.0 mm thick, being each one photopolymerized for 40 s. The restored halves were bisected perpendicular to the resin/tooth interface, and the resulting fragments were carefully polished with #600- up to 4000-grit SIC papers and treated according to the following protocol: first, the resin/tooth interface was etched with a 37% phosphoric acid gel for 5 s, rinsed and the samples were ultra-sonicated for 10 min, thoroughly washed with distilled water and immediately immersed in 2.5% glutaraldehyde in 0.1 M sodium cacodylate buffer (Acros Organics, New Jersey, USA) at pH 7.4, for 12 h at 4° C. After fixation, the samples were rinsed with 0.1 M sodium cacodylate buffer (Acros Organics, New Jersey, USA) several times, sequentially dehydrated in an ascending ethanol series (25% for 20 min; 50% for 20 min; 75% for 20 min; 90% for 30 min; 100% for 60 min), then immersed in 100% hexamethyldisizilane (HMDS) (Acros Organics, New Jersey, USA) for 10 min, placed on absorbing paper inside glass plates and left drying in an exhaust system. Specimens were mounted on stubs with their treated surfaces up-faced and sputter-coated with gold. The enamel and dentin surfaces and adhesive interfaces were examined with a JSM T330 scanning electron microscope (JEOL, Japan) operating at 15 kV, as regards the surface morphology provided by different treatments, and formation or not of a hybrid layer, focusing on its integrity, homogeneity and continuity along the interface, as well as on the arrangement, uniformity of size and characteristics of hybridization of resin tags.

The data were analyzed by visual and qualitative comparison of the surfaces and the interfaces.

Results

The analysis of the SEM micrographs revealed that phosphoric acid application on enamel led to the formation of microporosities, the peripheral tissues of the prisms were dissolved and the cores were intact (Type 2 pattern) (Fig. 1A). In dentin it was noticed presence of open tubules (arrows) and absence of smear layer; distinction between peritubular and intertubular dentin was provided by different contrasts (Fig. 1B). Analysis of the adhesive/enamel interface disclosed the presence of a hybrid layer with resinous tags (asterisk) (Fig. 1C) and at the adhesive/dentin interface it was observed clear formation of a homogenous hybrid layer with thickened tags in the superficial peritubular part (asterisks), as well as tags deeper than 10 μm from the surface (Fig. 1D).

On samples treated with air-abrasion, the enamel exhibited an irregular aspect and absence of a definite etch pattern (Fig. 1E). In dentin, fissures on the surface and occluded tubules (arrows) were observed (Fig. 1F). On the adhesive/enamel interface, the analysis disclosed no resinous tags, apart from the existence of gaps (arrows) on the interface (Fig. 2A). The dentin/adhesive interface showed no hybrid layer or tags (arrows) and, in larger magnifications, presence of aluminum oxide particles (asterisks) on the interface and fissure formation were observed (Fig. 2B).

The treatment with air-abrasion and subsequent phosphoric acid application produced on enamel a surface pattern similar to the Group I (acid etching solely) (Fig. 2C). In dentin, superficial fissures, open tubules and surface irregularities exposing peritubular and intertubular dentin were registered (Fig. 2D). Analysis of the adhesive/enamel and adhesive/dentin interfaces showed formation of hybrid layer and presence of tags; the tags displayed conical form at the mouth of the dentine tubules (asterisks) (Fig. 2E, F).

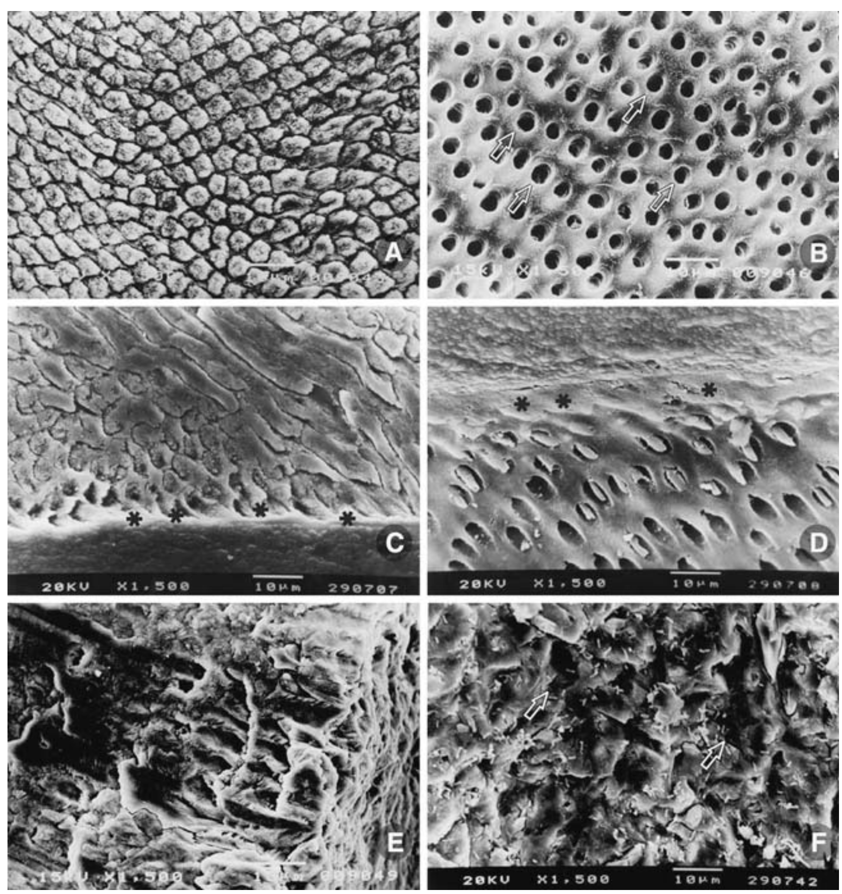

Fig. 1 (**A**) Enamel etched with 37% phosphoric acid—surface topography (×1500). (**B**) Dentin etched with 37% phosphoric acid—surface topography (×1500). (**C**) Morphology of adhesive/enamel interface after etching with 37% phosphoric acid (×1500). (**D**) Morphology of adhesive/dentin interface after etching with 37% phosphoric acid (×1500). (**E**) Enamel surface topography after treatment with air-abrasion (×1500). (**F**) Dentin surface topography after treatment with air-abrasion (×1500)

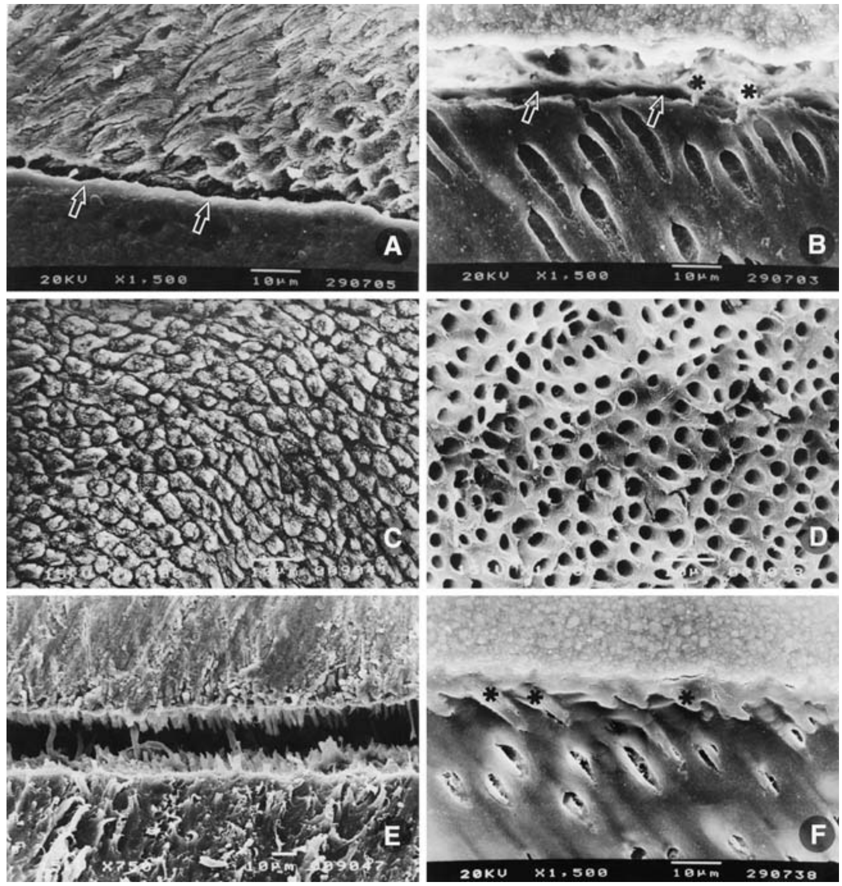

Fig. 2 (**A**) Morphology of adhesive/enamel interface after treatment with air-abrasion (×1500). (**B**) Morphology of adhesive/dentin interface after treatment with air-abrasion (×1500). (**C**) Enamel surface topography after treatment with air-abrasion followed by 37% phosphoric acid application (×1500). (**D**) Dentin surface topography after treatment with air-abrasion followed by 37% phosphoric acid application (×1500). (**E**) Morphology of adhesive/enamel interface after treatment with air-abrasion followed by 37% phosphoric acid application (×750). (**F**) Morphology of adhesive/dentin interface obtained after treatment with air-abrasion followed by 37% phosphoric acid application (×1500)

Discussion

Bonding of composite resin to enamel and dentin is one of the most important elements of modern dentistry. At least concerning the dentin, the reliability and durability of the adhesive interface still needs to be improved.

Acid etching as a method of enamel and dentin surfaces pretreatment has been widely accepted in restorative procedures [1]. The adhesion of resin to etched enamel is clearly due to micromechanical retention caused by resin tag formation and penetration into the spaces left by acid etching [35]. Air-abrasion procedures creates irregularities in the enamel that could enhance the mechanical interlocking effect, increasing the surface area and therefore, increasing the total surface energy [37]. However, the effectiveness of this technique is controversial, with some researchers supporting the etching ability of air-abrasion to enamel and/or dentin [38, 39], while others researchers deny its efficacy [40–42]. SEM observations of air-abraded enamel and dentin surfaces have shown that the surface irregularities increases with air-abrasive treatment; on the other hand, the characteristics of air-abraded enamel or dentin surfaces are much different from those treated chemically with phosphoric acid [43]. To improve the adhesive performance assessing bond strength results, subsequent etching of enamel and dentin is advocated by several authors [15, 44–46]. It is reported in the current literature that the air-abraded enamel surfaces are irregular but not similar to the honeycomb-like structure seen after acid etching [47]. In the conducted study, enamel surface treated by air-abrasion provided an irregular surface, absence of a definite etch pattern, and aluminum oxide particles on the air-abraded surface. These particles probably had a deleterious influence on the penetration of the adhesive system.

In the air-abraded dentin, tubules could not be observed, which suggests that the air-abrasive process creates a smear layer, and studies indicate that this layer must be removed for maximum dentin bond strengths [39, 43–45]. Factors such as smear layer, dentin tubule density, size, length, and sclerosis play significant roles in dentin bonding [48]. The characteristic of the smear layer affects bonds to dentin with resin bonding agents [49]. Most dentin bonding agents are dependent on smear layer removal and formation of the hybrid layer [3, 5]. Air-abraded dentin is quite different in appearance from chemically treated dentin. Dentinal tubules are occluded, but the surface does not have the familiar ‘‘smeared’’ appearance [48]. This study indicates that this layer must be removed for the formation of a well-defined resin infiltrated zone.

The analysis of the dentin surface revealed occluded dentin tubules, thereby providing a surface without a definite pattern and superficial fissures, which might have hampered the formation of a hybrid layer and favored the arise of gaps that were disclosed by the interface analysis. Distinctly from the treatment by air-abrasion solely, which provided lower penetration of the adhesive system, its combination to acid etching in the conducted study provided the formation of a hybrid layer with tags. This difference might be explained by the reduction or absence of smear layer after treatment with phosphoric acid. Therefore, it may be assumed that the etching gel might have penetrate deeper in the absence of (or presence of a very thin) smear layer [50], within the same period of time which the acid had to overcome the obstacle of smear layer before it could reach and interact as deep with the underlying dentin.Within the frame of this study, even though there are some controversial points related to the morphological aspects of the dental surface after air-abrasion application, the use of this technology requires compulsorily the subsequent acid conditioning in order to obtain a homogenous hybrid layer with uniform tags. In summary, although more research is needed to improve air-abrasion application, it does not appear that air-abrasion technology without subsequent acid etching provides a significant advantage over traditional tooth surface treatment methods and, in fact, appears to be inferior to the acid etching technique for use in adhesive restorative procedures.

Conclusions

Based on the findings of the reported research and within the limitations of an in vitro study, it may be concluded that:

1. Application of air-abrasion solely did not produce appropriate surfaces for adhesion and adversely affected the interaction pattern of the adhesive system with the substrates.
2. Treatment with air-abrasion followed by acid etching provided tooth surface patterns similar to the acid etching solely and this combination provided the formation of consistent resin-enamel and resin-dentin hybridization zones.

Acknowledgments This study was supported by FAPESP (Fundação de Amparo à Pesquisa do Estado de São Paulo)-Grant # 01/01183-4.

References

1. M. BUONOCORE, *J. Dent. Res.* **35** (1955) 849
2. D. H. PASHLEY and R. M. CARVALHO, *J. Dent.* **25** (1997) 355
3. N. MOSZNER, U. SALZ and J. ZIMMERMANN, *Dent. Mater.* **21** (2005) 895
4. B. VAN MEERBEEK, A. DHEM, M. GORET-NICAISE, M. BRAEM, P. LAMBRECHTS and G. VANHERLE, *J. Dent. Res.* **72** (1993) 495
5. N. NAKABAYASHI, M. ASHIZAWA, M. NAKAMURA, *Quintessence Int.* **23** (1992) 135
6. A. J. GWINNETT, *Am. J. Dent.* **6** (1993) 7
7. R. B. BLACK, *J. Am. Dent. Assoc.* **32** (1945) 955
8. R. B. BLACK, *J. Am. Dent. Assoc.* **41** (1950) 701
9. R. B. BLACK, *J. Am. Dent. Assoc.* **50** (1955) 408
10. K. A. LAURELL and J. A. HESS, *Quintessence Int.* **26** (1995) 139
11. O. AYSEGUL, O. NURHAN, B. HALUK and T. DILEK, *Oper. Dent.* **30** (2005) 164
12. L. REIS, M. A. CHINELATTI, S. A. M. CORONA, R. G. PALMA-DIBB and M. C. BORSATTO, *J. Mater. Sci. Mater. Med.* **15** (2004) 1213
13. S. A. M. CORONA, M. C. BORSATTO, R. G. PALMA-DIBB, R. P. RAMOS, A. BRUGNERA Jr and J. D. PECORA, *Oper. Dent.* **26** (2001) 491
14. R. GUIRGUIS, J. LEE and J. CONRY, *Pediatr. Dent.* **21** (1999) 311
15. L. LUPI-PEGURIER, M. MULLER-BOLLA, M. F. BERTRAND, T. FRADET and M. BOLLA, *J. Adhes. Dent.* **6** (2004) 43
16. M. E. KATORA, T. JUBACH and M. M. POLIMUS, *Quintessence Int.* **9** (1981) 967
17. T. BROSH, S. STROUTHOU and O. SARNE, *J. Dent.* **99** (2005) 99
18. E. A. BERRY and M. WARD, *Quintessence Int.* **26** (1995) 559
19. M. E. OLSEN, S. E. BISHARA, P. DAMON and J. R. JAKOBSEN, *Am. J. Orthod. Dentofacial Orthop.* **112** (1997) 502
20. M. C. BORSATTO, S. A. M. CORONA, A. B. E. CATIRSE, R. G. PALMA-DIBB, T. N. NASCIMENTO and R. A. S. S. ROCHA, *Braz. Dent. J.* **13** (2002) 175
21. F. AHID, M. F. ANDRADE, E. A. CAMPOS, F. LUSCINO and L. G. VAZ, *Am. J. Dent.* **17** (2004) 447
22. S. A. LOS and W. W. BARKMEIER, *Oper. Dent.* **19** (1994) 169
23. R. B. BESTER, F. A. WET and J. C. NEL, *Int. J. Prosthodont.* **8** (1995) 46
24. R. E. GOLDSTEIN and F. M. PARKINS, *J. Am. Dent. Assoc.* **126** (1995) 761
25. A. BROSTEK, *Oral Health Prev. Dent.* **2** (2004) 313
26. C. DUQUE, R. C. SILVA and L. SANTOS-PINTO, *J. Clin. Pediatr. Dent.* **29** (2004) 5
27. P. LAMBRECHTS, D. MATTAR, J. de MUNCK, L. BERGMANS, M. PEUMANS, G. VANHERLE and B. VAN MEERBEEK, *J. Esthet. Restorative Dent.* **14** (2002) 167
28. A. R. YAZICI, G. OZGUNALTAY and B. DAYANGAC, *Oper. Dent.* **27** (2002) 360
29. Z. C. ÇEHRELI, A. R. YAZICI, T. AKCA and G. ÖZGÜNALTAY, *J. Dent.* **31** (2003) 429
30. K. FERDIANAKIS, *J. Clin. Pediatr. Dent.* **22** (1998) 221
31. S. HATIBOVIC-KOFMAN, G. Z. WRIGHT and I. BRAVERMAN, *Pediatr. Dent.* **20** (1998) 173
32. D. C. N. CHAN, J. B. SUMMITT, F. GARCÍA-GODOY, T. J. HILTON and K. H. CHUNG, *Oper. Dent.* **24** (1999) 331
33. R. W. ELLIS, M. LATTA and G. H. WESTERMA, *Am. Acad. Pediatr. Dent.* **21** (1999) 316
34. C. PERUCHI, L. SANTOS-PINTO, A. SANTOS-PINTO, E. BARBOSA E SILVA, *Quintessence Int.* **33** (2002) 279
35. A. J. GWINNETT and A. MATSUI, *Arch. Oral Biol.* **12** (1995) 1615
36. R. OSORIO, M. TOLEDANO, M. A. CABRERIZO-VILCHEZ, J. I. ROSALES, M. BRAVO and E. OSORIO, *J. Biomed. Mater. Res. Part B* **33** (1996) 269
37. R. D. PHOENIX and C. SHEN, *Int. J. Prosthodont.* **8** (1995) 187
38. A. MUJDECI and O. GOKAY, *J. Prosthet. Dent.* **92** (2004) 245

39. H. KAJIHARA, S. SUZUKI, Y. MINESAKI, H. KURASHIGE and T. TANAKA, *Am. J. Dent.* **17** (2004) 161
40. J. MANHART, K. C. HUTH, H. Y. CHEN and R. HICKEL, *Am. J. Dent.* **17** (2004) 12
41. L. C. CASTRO and A. C. GALVÃO, *J. Clin. Pediatr. Dent.* **28** (2004) 249
42. D. DUANGTHIP and A. LUSSI, *Pediatr. Dent.* **25** (2003) 527
43. T. NIKAIDO, M. KATAUMI, M. F. BURROW, S. INOKOSHI, T. YAMADA and T. TAKATSU, *Oper. Dent.* **21** (1996) 218
44. L. B. ROEDER, E. A. BERRY, C. YOU and J. M. POWERS, *Oper. Dent.* **20** (1995) 186
45. J. MANHART, A. MEHL, R. SCHROETER, B. OBSTER and R. HICKEL, *Oper. Dent.* **24** (1999) 223
46. W. L. W. HOGERVORST, A. J. FEILZER and B. PRAHL-ANDERSEN, *Am. J. Orthod. Dentofacial Orthop.* **117** (2000) 20
47. K. HOTTA, M. MOGI, F. MIURA and N. NAKABAYASHI, *Dent. Mater.* **8** (1992) 173
48. H. O. HEYMANN and S. C. BAYNE, *J. Am. Dent. Assoc.* **124** (1993) 26
49. L. TAO, D. H. PASHLEY and L. BOYD, *Dent. Mater.* **4** (1988) 208
50. J. PERDIGÃO, S. EIRIKSSON and B. T. ROSA, *Quintessence Int.* **32** (2001) 142