DOI: 10.3109/00016357.2014.971868

Acta Odontologica Scandinavica. 2015; 73: 176–181

informa healthcare

ORIGINAL ARTICLE

Effect of thermocycling on roughness of nanofill, microfill and microhybrid composites

PAULO HENRIQUE DOS SANTOS[1], ANDERSON CATELAN[2], ANA PAULA ALBUQUERQUE GUEDES[3], THAÍS YUMI UMEDA SUZUKI[1], ANDRÉ GUSTAVO DE LIMA GODAS[3], ANDRÉ LUIZ FRAGA BRISO[3] & ANA KARINA BEDRAN-RUSSO[4]

[1]*Department of Dental Materials and Prosthodontics, Araçatuba School of Dentistry, São Paulo State University, Araçatuba, SP, Brazil,* [2]*Department of Restorative Dentistry, Piracicaba Dental School, University of Campinas, Piracicaba, SP, Brazil,* [3]*Department of Restorative Dentistry, Araçatuba School of Dentistry, São Paulo State University, Araçatuba, SP, Brazil, and* [4]*Department of Restorative Dentistry, College of Dentistry, University of Illinois at Chicago, Chicago, IL, USA*

Abstract

Objective. This study aimed to investigate the surface roughness of composite resins subjected to thermal cycles procedure. ***Materials and methods.*** Two microfill, four microhybrid and four nanofill composites were used. The surface roughness (Ra) was initially measured in a profilometer using a cut-off 0f 0.25 mm, after 3000 and 10,000 thermal cycles. Data were subjected to ANOVA and Fischer's test (α = 0.05). ***Results.*** Overall, 3000 thermal cycles increased the surface roughness values for all materials and there was a trend in all groups to decrease the roughness after 10,000 thermal cycles. ***Conclusions.*** The composition of material, including the type of organic matrix, could be more relevant to roughness maintenance over time than the general behavior of composites based on particles fillers. The maintenance of smooth surface in resin-based composite restorations is totally dependent of organic composition of the material.

Key Words: *roughness, composite resin, degradation.*

Introduction

Since the beginning of the 1960s [1], composite resin has been available as an esthetic material for restorative procedures. The use as a posterior restorative material has been substantially increased over the last few years [2]. This material consists of a resin matrix and filler particles that are chemically linked by silane coupling agents. Several composite materials are available for direct dental restorations, comprising microhybrid, microfilled and nanofilled composites [3].

Differences among composite resins such as the monomer system, filler composition and matrix-filler coupling chemistry may result in different mechanical degradation [3]. It has been reported that damage in composite may result from deterioration of matrix, which may reduce the survival probability of polymer restorations *in vivo* [3]. The structure of resin matrix, coupling agent and the characteristics of filler particles have a direct impact on the surface smoothness of composite resins [4]. The type of inorganic particles, size and extend of filler loading are considered the most important factors [4].

Surface roughness of restorative materials has been recognized as a parameter of high clinical relevance for plaque accumulation, staining susceptibility and wear [5,6]. If the restoration has a surface roughness of 0.2 µm (Ra) or more, dental plaque accumulation may increase the risk for both caries and periodontal inflammation [7]. The effective finishing and polishing procedures during the restorative process would improve the surface smoothness and compensate for surface roughness generated by wear mechanisms on restorations [8].

The mechanical properties of composite resin can be affected by hydrolytic degradation [9]. In *in vitro*

Correspondence: Professor Paulo dos Santos, Department of Dental Materials and Prosthodontics, Araçatuba School of Dentistry, UNESP, R. José Bonifácio, 1193, Vl. Mendonça, Zip code: 16015-050 Araçatuba, SP, Brazil. Tel: +55 18 3636 802. Fax: +55 18 3636 245. E-mail: paulosantos@foa.unesp.br

(Received 8 July 2014; accepted 14 September 2014)

ISSN 0001-6357 print/ISSN 1502-3850 online © 2014 Informa Healthcare
DOI: 10.3109/00016357.2014.971868

studies, the long-term water storage and thermal cycling are considered relevant conditions to test the durability of resin bonds [10]. Furthermore, roughness of some resin-based materials can be changed by the toothbrushing and thermocycling process [11] and could affect the durability of the composite restorations.

In this manner, the study of surface roughness of different resin-based materials as well as the effect of degradation process to this property is essential to provide longevity of esthetic restorations. Therefore, this study investigated the roughness of nanofill, microfill and microhybrid composite resins subjected to thermal cycles procedures. The research hypothesis is that the thermal cycling could affect differently the roughness of materials due to composition difference between composites such as type of filler and resinous matrix.

Materials and methods

The materials used in this study are show in Table I, including two microfill (Renamel Microfill, Cosmedent Inc., Chicago, IL; Durafill, Heraeus Kulzer Inc., Armonk, NY), four microhybrid (Point 4, Kerr Corp., Orange, CA; Filtek Z250, 3M ESPE, St. Paul, MN; Renamel Microhybrid, Cosmedent Inc; Clearfil AP-X, Kuraray Medical Inc. Okayama, Japan) and four nanofill (Filtek Supreme Plus, 3M ESPE; Premise, Kerr Corp.; Renamel Nano, Cosmedent Inc; Clearfil Majesty Esthetic, Kuraray Medical Inc.) composite resins. Ten samples of each resin-based composite were carried out using a polyethylene mold (2 mm in thickness and 6.2 mm in diameter), totaling 100 samples. A mylar strip was placed on the top and bottom of the molds and the cavity was completely filled with composite resin. A thin glass plate was placed over the composite and the samples were light-cured for 60 s using a quartz-tungsten-halogen curing unit (Optilux 501; Kerr/Demetron, Danbury, CT) at 450 mW/cm^2, monitored by a radiometer (model 100; Kerr/Demetron).

All samples were then finished and polished process using the Top Finisher polishing system (Cosmedent Inc.) following the sequence: Flexi-discs (coarse, medium, fine and superfine), Porcelize and Enamelize polishing paste with Flexibuff disks (Cosmedent Inc.), using a low-speed handpiece (25,000 rpm).

The surface roughness (Ra) was measured in a perfilometer SJ-401 (Mitutoyo Corp., Tokyo, Japan). The Ra value was used because it represents the arithmetical mean of roughness of a surface and is the parameter most used for this purpose. Each measurement was obtained after turning the sample 120°, totaling three measurements using a cut-off of 0.25 mm. The samples were then stored in distilled water at 37°C until the thermal cycling procedure begins.

Table I. Materials, manufacturer, batch number and composition of composite resins.

Material	Manufacturer	Batch #	Composition
Renamel Microfill	Cosmedent Inc.	064329 K	BisGMA, BisEMA, 60 wt% pyrogenic silica acid filler of 0.02–0.04 μm
Durafill	Heraeus Kulzer	010203	BisGMA, UDMA, TEGDMA, SiO_2 filler (0.02–0.07 μm), pre-polymerized filler (10–20 μm)
Point 4	Kerr Corp.	2753914	BisGMA, TEGDMA, BisEMA, 76 wt% and 57 vol%. Barium and silica glass filler (0.4 μm)
Filtek Z250	3M ESPE	8EC	BisGMA, UDMA, BisEMA, camphorquinone, inorganic filler zirconia/silica 60 vol% (0.01–3.5 μm)
Renamel Microhybrid	Cosmedent Inc.	073013 J	Multifunctional acrylic resins and inorganic fillers (0.04–3 μm, 75 wt% and 56 vol%
Clearfil AP-X	Kuraray Medical Inc.	00983B	Bis-GMA, TEGDMA, silanized barium glass filler, silanized silica filler, silanized colloidal silica, camphorquinone, catalysts, accelerators, pigments
Filtek Supreme Plus	3M ESPE	8PX	Nanosilica filler (0.02 μm), zirconia/silica nanoclusters aggregates (0.6–1.4 μm), BisGMA, BisEMA, UDMA, TEGDMA
Premise	Kerr Corp.	2753951	Barium glass (0.4 μm), non-agglomerated silica nanoparticles (0.02 μm), pre-polymerized filler, etoxylated BisEMA, TEGDMA
Renamel Nano	Cosmedent Inc.	073621 C	Non-agglomerated nanosilica (20 nm), agglomerated nanosilica silanized (40 nm), Ba-Al-Fluro-Borsilica glass silanized (<0.7 μm), BDDMA, BisGMA, UDMA
Clearfil Majesty Esthetic	Kuraray Medical Inc.	00011A	Silanized barium glass filler; pre-polymerized organic filler, hydrophobic aromatic dimethacrylate, BisGMA, camphorquinone

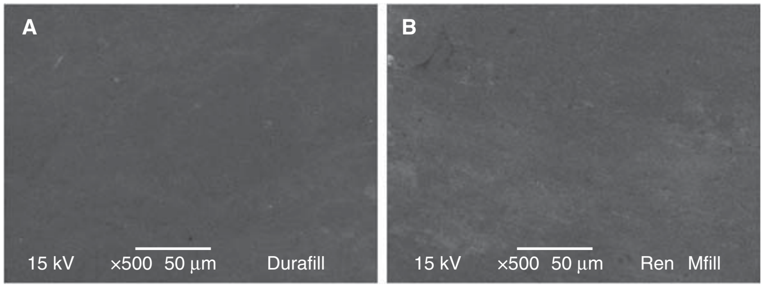

Figure 1. Microfill composites after thermal cycling procedure: (A) Durafill; (B) Renamel microfill.

Thermal cycling (alternate immersion of samples in distilled water with a temperature of 5 and 55°C, 5 min each and transfer interval of 5 s) was carried out in a thermal cycler MSTC-3 Plus thermal cycling machine (ElQuip, São Carlos, SP, Brazil). Roughness measurements were collected after 3000 and 10,000 thermal cycles. The data were submitted to repeated measures ANOVA and Fisher's PLSD test ($p < 0.05$).

Selected samples from each experimental group were mounted on metal stubs, gold-sputter coated (SCD 050, Bal-Tec AG, Balzers, Liechtenstein) and surface morphology was evaluated under a scanning electron microscopy (SEM) (JSM-5600LV, JEOL, Tokyo, Japan), under magnification of 500×.

Results

Table II shows the surface roughness of composite resins before and after 3000 and 10,000 thermal cycles. After 3000 cycles there was an increase on the surface roughness for all materials, except for Filtek Z250 and Renamel Nano ($p \leq 0.05$). After 10,000 cycles there was a trend of decreasing values of surface roughness, with no statistically significant difference for the initial measurements, except for some microfill composites (Renamel Microfill and Durafill) and for Clearfil AP-X ($p \leq 0.05$).

The Renamel Microfill showed less values of surface roughness than Durafill before and after 10,000 cycles ($p < 0.05$). There is no difference between the materials after 3000 cycles ($p = 0.854$). In Figure 1, it seems that, for both microfill composites, there are no protruded fillers in the materials surface. No evidence of worn is observed in the SEM images.

Clearfil AP-X showed the highest values of surface roughness in all measurements when compared to all other microhybrid composites ($p \leq 0.05$), regardless of thermal cycling procedure. In Figure 2, some fillers are exposed on the surface of Clearfil AP-X after a thermal cycling procedure (Figure 2B) compared to other microhybrid composites, which seem to have a more smooth surface (Figures 2A, C and D). There was no statistically significant difference among the other materials before and after 3000 and 10,000 thermal cycles ($p > 0.05$).

There is no difference among the materials before and after 10,000 thermal cycles ($p \leq 0.05$). After 3000 thermal cycles, the Renamel Nano showed

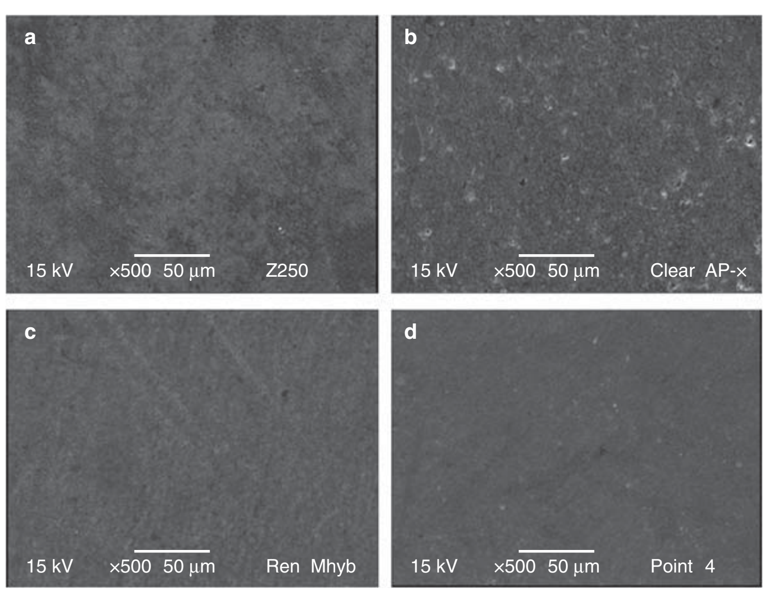

Figure 2. Microhybrid composites after thermal cycling procedure: (A) Filtek Z250; (B) Clearfil AP-X; (C) Renamel mycrohybrid; (D) Point 4.

the lowest surface roughness values (0.06 μm) when compared to Premise (0.10 μm) and Clearfil Majesty Esthetic (0.11 μm). There is no difference between Renamel Nano and Filtek Supreme Plus after 3000 thermal cycles ($p = 0.150$). Even though some filler could be found in the nanofill composites (Figure 3), there is no evidence of exposure of them after thermocycling procedure.

Discussion

The findings of this *in vitro* study indicate that thermal cycling have a critical effect on surface roughness of composite resins, regardless of the filler composition. Therefore, the research hypothesis was accepted. It could be observed that most resin-based materials showed an increase in roughness values after 3000 thermal cycles. A previous study also showed that thermal cycling significantly affected the surface texture of composites with dislodgement of filler particles [12]. However, a correlation between thermocycling and clinical longevity of dental composites is difficult due to the varied cycles number, different temperatures, dwell time and intervals between baths used in the studies.

Thermocycled samples have been subjected to temperature fluctuations, generating thermal stresses and leading to microcracks in the matrix or failure at the filler/matrix interface [13]. Moreover, exposure to water may cause hydrolytic degradation of filler's silane coating or swelling of the matrix [9,13]. Differences in filler exposure after thermal cycling are thus most likely due to matrix degradation, leading to exposure of underlying filler particles and an increased roughness, as observed after 3000 cycles for most of materials tested. Composites containing hydrophilic components, like TEGDMA or TEGMA as a matrix component, may be more susceptible to matrix degradation [13,14], because they allow water to penetrate more easily due to its hydrophobicity. Filtek Z250 and Renamel Nano were not affected by thermal cycling (Table I). These composites do not contain in their compositions hydrophilic components, which could be in part be responsible for these results.

There was a trend in all groups to decrease the surface roughness after 10,000 thermal cycles compared to 3000 thermal cycles, except for some microfill composites (Durafill and Renamel Microfill) and Clearfill AP-X (Figures 1 and 2, respectively). The size, hardness and amount of filler dictate the surface roughness of composite; which enhance the mechanical properties of the resin-based composites [15,16]. A study showed that composite wears depth decreased monotonically with increase of filler level [17]. As the microfill composites have less content of particle fillers in its composition, they probably have been more significantly affected by increased thermal cycles (Figure 1). A previous study [18] reported higher roughness values for Clearfil AP-X, caused by the largest filler size and irregularity of particles compared with other restorative composites (Figure 2). Moreover, this material has a TEGDMA hydrophilic component, which is susceptible to hydrolytic degradation [13,14]. However, despite the surface roughness being influenced by the amount and size of filler, this study showed that the composition of material, including the type of organic matrix, could be more relevant to the preservation of roughness over time.

For the nanofill composites, all materials showed an increase in roughness values after 3000 thermal cycles compared with initial measurements, except for Renamel Nano (Table II). The 82% of fillers in composition of materials including agglomerated and non-agglomerated nanofillers could account for these results. The surface appearance of the nanofill composites after thermocycling is shown in Figure 3. All nanofill composites showed similar roughness after 10,000 thermal cycles. Even though some

Table II. Surface roughness (Ra, μm) means (SD) of the resin-based composites before and after thermal cycling.

Material	Initial	3000 cycles	10,000 cycles
Renamel Microfill	0.04 (0.01)^{a}	0.09 (0.02)^{c}	0.06 (0.01)^{b}
Durafill	0.07 (0.01)^{a}	0.11 (0.02)^{b}	0.11 (0.01)^{b}
Filtek Supreme Plus	0.07 (0.01)^{a}	0.10 (0.02)^{b}	0.07 (0.01)^{a}
Premise	0.06 (0.01)^{a}	0.11 (0.02)^{b}	0.06 (0.01)^{a}
Renamel Nano	0.07 (0.01)^{a}	0.06 (0.01)^{a}	0.07 (0.02)^{a}
Clearfil Majesty Esthetic	0.06 (0.01)^{a}	0.11 (0.01)^{b}	0.05 (0.01)^{a}
Point 4	0.04 (0.01)^{a}	0.09 (0.03)^{b}	0.04 (0.01)^{a}
Filtek Z250	0.06 (0.01)^{a}	0.06 (0.02)^{a}	0.06 (0.01)^{a}
Renamel Microhybrid	0.05 (0.01)^{a}	0.08 (0.01)^{b}	0.05 (0.01)^{a}
Clearfil AP-X	0.11 (0.01)^{a}	0.13 (0.02)^{b}	0.13 (0.02)^{b}

Lower case letter indicates statistical difference in the row ($p \leq 0.05$).

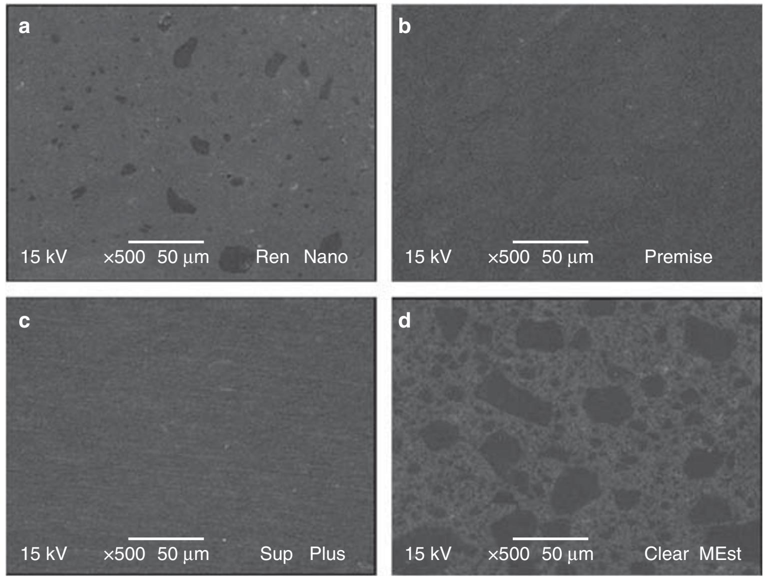

Figure 3. Nanofill composites after thermal cycling procedure: (A) Renamel Nano; (B) Premise; (C) Filtek Supreme Plus; (D) Clearfil Majesty Esthetic.

composites showed higher roughness values after thermocycling, especially after 3000 thermal cycles, all of them showed roughness under the limit proposed by the literature (0.2 μm) [7].

The surface roughness parameter (Ra) represents arithmetic average value of the departure from profile from centerline [11]. The increase on roughness after thermocycling procedures might cause several problems such as surface stain, dental plaque accumulation and wear of occluding teeth [5,11]. Furthermore, organic matrices of composites would have absorbed some water [11], causing hygroscopic expansion in resinous matrix and filler phase, thereby enhancing the weakening of matrix–filler interface [12,19]. Data from this study demonstrated some of the changes caused by thermocycling in direct restorative materials. However, further research is needed to assess simultaneously the effect of other degradation processes such as erosion and mechanical abrasion.

Within the limitations of this *in vitro* study, the following can be concluded: (1) overall, the thermal cycling (3000 cycles) increased roughness values for all materials; (2) there was a trend in all groups to decrease surface roughness after 10,000 thermal cycles; and (3) composition of material, including the type of organic matrix, could be more relevant to maintenance of roughness over time than general behavior of particles fillers.

Acknowledgments

The authors would like to thank the generous donations of materials by Cosmedent Inc., Heraeus Kulzer, Kerr Sybron, 3M ESPE, and Kuraray Medical Inc.

***Declaration of interest*:** The authors report no conflicts of interest. The authors alone are responsible for the content and writing of the paper.

References

[1] Bowen RL. Properties of a silica-reinforced polymer for dental restorations. J Am Dent Assoc 1963;66:57–64.

[2] dos Santos PH, Pavan S, Assunção WG, Consani S, Correr-Sobrinho L, Sinhoreti MA. Influence of surface sealants on microleakage of composite resin restorations. J Dent Child 2008;75:24–8.

[3] Hahnel S, Henrich A, Bürgers R, Handel G, Rosentritt M. Investigation of mechanical properties of modern dental composites after artificial aging for one year. Oper Dent 2010;35: 412–19.

[4] Marghalani HY. Effect of filler particles on surface roughness of experimental composite series. J Appl Oral Sci 2010;18:59–67.

[5] Aykent F, Yondem I, Ozyesil AG, Gunal SK, Avunduk MC, Ozkan S. Effect of different finishing techniniques for restorative materials on surface roughness and bacterial adhesion. J Prosthet Dent 2010;103:221–7.

[6] Correr GM, Alonso RC, Baratto-Filho F, Correr-Sobrinho L, Sinhoreti MA, Puppin-Rontani RM. *In vitro* long-term degradation of aesthetic restorative materials in food-simulating media. Acta Odontol Scand 2012;70:101–8.

[7] Bollen CML, Lambrechts P, Quirynen M. Comparison of surface roughness of oral hard materials to the threshold surface roughness for bacterial plaque retention: a review of the literature. Dent Mater 1997;13:258–69.

[8] Marghalani HY. Effect of finishing/polishing systems on the surface roughness of novel posterior composites. J Esthet Restor Dent 2010;22:127–38.

[9] Chadwick RG. Thermocycling - the effects upon the compressive strength and abrasion resistance of three composites. J Oral Rehabil 1994;21:533–43.

[10] Lüthy H, Loeffel O, Hammerle CH. Effect of thermocycling on bond strength of luting cements to zirconia ceramics. Dent Mater 2006;22:195–200.

[11] Cho LR, Yi YJ, Heo SJ. Effect of tooth brushing and thermal cycling on a surface change of ceromers finished with different methods. J Oral Rehabil 2002;29:816–22.

[12] Minami H, Hori S, Kurashige H, Murahara S, Muraguchi K, Minesaki Y, et al. Effect of thermal cycling on surface texture of restorative composite materials. Dent Mater J 2007;26:316–22.

[13] Rinastiti M, Ozcan M, Siswomihardjo W, Busscher HJ. Effects of surface conditioning on repair bond strengths of non aged and aged microhybrid, nanohybrid, and nanofilled composite resins. Clin Oral Investig 2011;15:625–33.

[14] McCabe JF, Rusby S. Water absorption, dimensional change and radial pressure in resin matrix dental restorative materials. Biomaterials 2004;25:4001–7.

[15] Gedik R, Hürmüzlü F, Coskun A, Bektas OO, Özdemir AK. Surface roughness of new microhybrid resin-based composites. J Am Dent Assoc 2005;136:1106–12.

[16] Endo T, Finger WJ, Kanehira M, Utterodt A, Komatsu M. Surface texture and roughness of polished nanofill and nanohybrid resin composites. Dent Mater J 2010;29:213–23.

[17] Xu HH, Eichmiller FC, Smith DT, Schumacher GE, Giuseppetti AA, Antonucci JM. Effect of thermal cycling on whisker-reinforced dental resin composites. J Mater Sci Mater Med 2002;13:875–83.

[18] Senawongse P, Pongprueska P. Surface roughness of nanofill and nanohybrid resin composites after polishing and brushing. J Esthet Restor Dent 2007;19:265–73.

[19] Tuncer S, Demirce M, Tiryaki M, Unlü N, Uysal Ö. The effect of a modeling resin and thermocycling on the surface hardness, roughness, and color of different resin composites. J Esthet Restor Dent 2013;25:404–19.