DENTAL MATERIALS 27 (2011) 173–179

available at www.sciencedirect.com

ScienceDirect

journal homepage: www.intl.elsevierhealth.com/journals/dema

Surface integrity of solvent-challenged ormocer-matrix composite

Larissa Maria Cavalcante[a,b,*], *Luis Felipe J. Schneider*[a,b], *Nick Silikas*[a], *David C. Watts*[a]

[a] *Biomaterials Research Group, The University of Manchester, School of Dentistry, UK*
[b] *School of Dentistry, Federal Fluminense University, Niterói, RJ, Brazil*

ARTICLE INFO

Article history:
Received 22 February 2010
Received in revised form
9 September 2010
Accepted 12 October 2010

Keywords:
Knoop hardness
Solvent-challenge
Surface integrity
Ethanol
Ormocer-based material
Dimethacrylate-based material
Nanohybrid composites

ABSTRACT

Objective. To investigate the surface integrity of solvent-challenged ormocer-matrix composites, photoactivated by different light exposure modes, through surface-hardness measurements at different periods of time; and to compare such behavior with dimethacrylate-based materials.

Methods. One hundred percent ormocer-based matrix (experimental ormocer (ORM)), a commercial mixed dimethacrylate-ormocer-based matrix (Admira (ADR)) and two commercial dimethacrylate-based matrix composites (experimental controls, (Grandio (GRD) and Premise (PRE)) were evaluated. Disk specimens (4 mm × 2 mm) were prepared from each material and light-activated using either a standard (S) or soft-start (SS) light exposure protocol with an LED-curing unit. Top, irradiated surface Knoop hardness (KHN) was measured within the following experimental groups ($n = 5$): Group 1: immediately after exposure; Group 2: after dry and dark storage, Group 3: after storage in distilled water, and Group 4: immersion in absolute ethanol. Hardness of Groups 2–4 were measured after 7 days storage. Immediate hardness values were submitted to Student's *t*-tests separately for each material. Hardness values after treatments were submitted to two-way ANOVA and Tukey's post hoc test to compare values among different storage media and light exposure mode protocols. Comparisons among materials were described using percentage of hardness change. Statistical testing was performed at a pre-set alpha of 0.05.

Results. Immediate hardness values were not affected by the light exposure mode, regardless of the material. In general, exposure mode did not significantly affect hardness after 7 days storage, regardless of storage media or material. After 7 days dry storage, hardness values increased for all materials relative to immediate testing, and decreased after water and ethanol storage, with ethanol showing the greatest effect. The experimental ormocer-based material had the lowest percentage hardness change and thus proved more resistant to solvent degradation than the other materials, regardless of the light exposure method.

Significance. Irradiated surface hardness values and surface integrity were unaffected by light exposure mode, regardless of the material tested. The experimental ormocer-based material presented the least change in hardness as a result of solvent challenge than any of the commercial products: ormocer or conventional resin-based, and thus showed better surface integrity.

* *Corresponding author at*: Faculdade de Odontologia da Universidade Federal Fluminense, Campus do Valonguinho, Rua Mário Santos Braga, n°30/3° andar, Centro, CEP 24020-140, Niterói, RJ, Brazil. Tel.: +55 21 81033636.
E-mail address: lara_cavalcante@yahoo.com.br (L.M. Cavalcante).
0109-5641/$ – see front matter © 2010 Academy of Dental Materials. Published by Elsevier Ltd. All rights reserved.
doi:10.1016/j.dental.2010.10.002

1. Introduction

An ormocer-resin-based resin composite is a hybrid structure not generated by the classic means of physically mixing ground glass with a polymerizable methacrylate-based resin, but through special processing based on molecular scale technology. This technology combines organic and inorganic components at nanoscopic scale through the sol–gel method [1]. The main characteristic of this type of material is the linking of organic groups to the inorganic backbone, formed by hydrolysis and condensation of alkoxides. The organic component (organic polymer) is responsible for network formation, flexibility, and optical properties. The inorganic unit (glasses, ceramics) lowers thermal expansion as well as provides chemical and thermal stability [1,2].

Ormocers are successfully used in high-tech industries, such as optical coatings, electronics, and medical technology. In Dentistry, admixed (a material mixture of dimethacrylate and ormocer-organic matrix) and pure ormocer-matrix have been used to overcome drawbacks of conventional dimethacrylate-matrix composites, such as elution of monomers attributable to toxicity and to allergenic reactions [3–5]. Special, pure ormocer-matrix composites [6] are believed to offer enhanced clinical performance through high wear resistance, reduced polymerization shrinkage, enhanced biocompatibility, and long-lasting polymer-matrix stability [7,8]. According to manufacturer information, the new Ormocer formulation has no free monomers, thus eliminating the risk of allergic reaction. However the stability (surface integrity) of this new, experimental material (pure Ormocer-matrix) when challenged by fluids encountered in the oral environment (solvent challenge) is not well known.

Surface integrity influences the long-term clinical performance of resin-composite restorations and can be affected by material formulation and/or degradation due to exposure in the oral environment [9]. Surface characteristics are also influential in the material's resistance to load application and the generation of scratches [10]. Hardness is a surface characteristic that evaluates material's surface resistance to localized plastic deformation or scratching by penetration caused by a constant applied load [11]. Surface hardness can also be an indicator of durability, degree of conversion (DC), and crosslink density (CLD) [11–13].

The CLD of a polymer network can be indirectly assessed by repeated hardness measurements before and after the immersion in organic solvents [14–19]. It is generally accepted that highly cross-linked polymers are more resistant to degradation and to solvent uptake, whereas linear polymers present a less dense polymer network allowing solvent molecules to diffuse more readily [20]. By using this methodology, investigations have shown that polymers with similar DC could display distinctly different CLD due to differences in polymer network cross-linking [14,15] as a result of different rates of polymerization. Thus, elevated light irradiance levels would generate polymers with higher CLD than those using lower levels, which might produce a structure with less extensive CLD [14,15]. Light exposure protocols based on application of an initial low-level light irradiance, which are applied in hope to reduce polymerization shrinkage, could influence surface network formation, and the resulting surface integrity when challenged by fluids. However, this assumption is a matter of controversy [12,19,21–24].

Scarce information is available about the surface integrity of materials formulated with a wide range of compositions and submitted to different light exposure protocols. Furthermore, no data can be found related to the surface integrity of pure ormocer-based-matrix composites when challenged by various clinically relevant solvents.

The purpose of this study was to investigate the effect of light exposure protocols (standard or soft-start) on immediate microhardness values (an indirect evaluation of degree of conversion) of different materials, as well as their ability to maintain initial hardness values when treated to various solvents, or remaining dry for a period of 7 days. Thus the surface integrity (maintenance of initial hardness) will be investigated as a function of restorative materials and light exposure methods.

Three research hypotheses are tested. The first assumes that for all restorative materials initial (immediate) hardness would be greater when using continuous exposure than when applying a soft-start method. The second hypothesis was that surface hardness values would be higher for specimens exposed to continuous exposure (compared to soft-start) after 7 days of storage (dry, water and ethanol). The last hypothesis anticipates that the experimental, pure ormocer-based composite would demonstrate the least percentage change in hardness value (immediate to 7 days, within all three storage treatments) among the commercial restorative products tested.

2. Materials and methods

2.1. *Restorative materials*

Compositions and manufacturer information for each restorative material used are presented in Table 1. An experimental ormocer consisting of one hundred percent ormocer-matrix (ORMOCER, Voco (ORM)) as well as a commercial, mixed ormocer-dimethacrylate-matrix (Admira, Voco (ADR)) were used. Experimental controls consisted of two commercial, dimethacrylate-matrix composites (Grandio, Voco (GRD) and Premise, Kerr (PRE)).

2.2. *Light exposure protocols and samples' preparation*

A mono-wave (blue) light emitting diode (LED) unit was used (Elipar Freelight 2, 3M ESPE Dental Products, St Paul, MN, USA). Uncured paste of each material was placed into a cylindrical brass mould (2 mm height × 4 mm inner diameter) and, covered with a Mylar strip and pressed with a microscope slide to form a flat surface. The light unit was held rigidly in place and specimens were light-activated with the light guide positioned directly on the Mylar strip. Two different light exposure protocols were used: continuous (C) consisting of a single 40-s exposure at 1100 mW/cm^2, (total of 44 J/cm^2) or an internal, selectable soft-start exposure was applied, providing an initial low, increasing irradiance value for 10 s, and then an irradiance similar to that of the continuous exposure for the remain-

Table 1 – Materials studied including their resin-matrix composition, filler type and wt%.

Material	Type	Matrix	Fillers	Manufacturer
(ADR)	ORMOCER	ORMOCER[a] matrix with substantial addition of conventional dimethacrylate monomers Bis-GMA, UDMA, TEGDMA	78 wt% SiO_2 and glass-ceramic	Voco, Cuxhaven, Germany
(ORM)	ORMOCER	ORMOCER[a] matrix with no additional dimethacrylate monomers	87 wt% SiO_2 and glass-ceramic	Voco, Cuxhaven, Germany
(GRD)	Nanohybrid	Bis-GMA, UDMA, TEGDMA	87 wt% SiO_2 and glass-ceramic	Voco, Cuxhaven, Germany
(PRE)	Nanohybrid	Bis-EMA, TEGDMA	84 wt% SiO_2 and barium glass	Kerr Corporation, USA

[a] ORMOCERS are inorganic/organic hybrid structures incorporating covalently-bonded methacrylate end-groups for free-radical cross-linking developed by the Fraunhofer-Institute, Germany.

ing 30 s (total of 35.5 J/cm^2). Irradiance was measured using a hand-held dental curing radiometer (Model 200, Demetron Research Corporation, Danbury, CT, USA). Twenty specimens of each material were made for each light curing mode. After polymerization, the specimens were mounted in 1-in. diameter phenolic rings (Buehler, Lake Bluff, USA) and embedded in self-curing polystyrene resin (Castoglas Resin, Buehler, Lake Bluff, USA) before polishing. The embedded specimens were submitted to mechanical polishing in a grinding/polishing machine (Buehler, Lake Bluff, USA) with a sequence of 800- and 1200-grit SiC papers under continuous water cooling.

Samples were then randomly assigned to one of four groups ($n = 5$): Group 1: immediate hardness testing after exposure; Group 2: hardness determined 7 days after dry, dark storage at 37 °C (control group); Group 3: hardness obtained 7 days following immersion in distilled water at 37 °C; and Group 4: 7 days soaking in absolute ethanol (100%) at 37 °C.

2.2.1. *Surface hardness*

Knoop hardness values was measured on the irradiated surfaces using an indenter (Future Tech FM-700, Tokyo, Japan) under a load of 25 g applied for 20 s. Measurements were performed at five locations on each specimen, and the average value was recorded as the hardness for a given specimen.

2.2.2. *Statistical analyses*

It is not appropriate to directly compare absolute hardness values among products, only with a product between light exposure modes. Thus, for each material, Student's *t*-test was used to compare immediate hardness values between continuous or soft-start modes. A two-way ANOVA was applied to examine the effect of storage condition and exposure mode within a given restorative material, followed by Tukey's post hoc test for pair-wise comparisons. All statistical testing was performed using a pre-set alpha of 0.05. Hardness comparison among the different materials was performed by determining the percentage change in hardness for a given material between the immediate reading and the 7 days storage value. Statistics were not performed on this data.

3. Results

Fig. 1 presents immediate hardness values for all materials using either the continuous or soft-start methods. Statistical analysis revealed no significant difference in surface hardness related to use of either exposure protocol within any test material.

Panels in Fig. 2 display surface hardness of each material after the various storage conditions for each light exposure protocol. For reference, immediate hardness values are also displayed. The results of the two-way ANOVA indicated a significant effect ($p < 0.001$) of storage condition and a non-significant effect of exposure mode (Admira $p = 0.058$, ORMOCER $p = 0.240$, Grandio $p = 0.026$ and Premise $p = 0.843$). The interaction term (storage condition × exposure mode) was not significant (Admira $p = 0.06$, ORMOCER $p = 0.403$, Grandio $p = 0.166$ and Premise $p = 0.150$). Regardless of material, the dry and dark storage produced the highest hardness values, followed, in sequence, by water, and then by ethanol. Furthermore, regardless of material and storage condition, there were no differences in hardness produced by either exposure mode.

Table 2 details the percentage of Knoop hardness changes (relative to the immediate value) of specimens stored in various solvents and photopolymerized using different exposure modes. As it can be seen, the outcomes follow the same pattern for standard and soft-start exposure mode. There was an increase of KHN values after 1 week in dark (dry) storage, how-

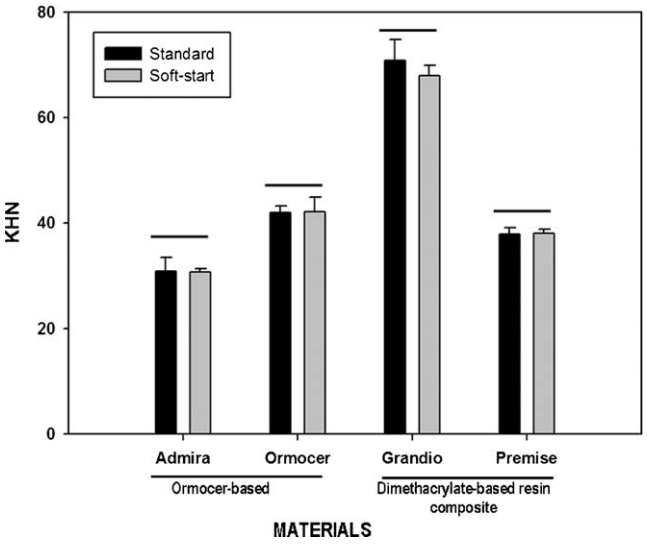

Fig. 1 – Initial Knoop hardness, for all materials tested, when using standard and soft-start light curing protocols.

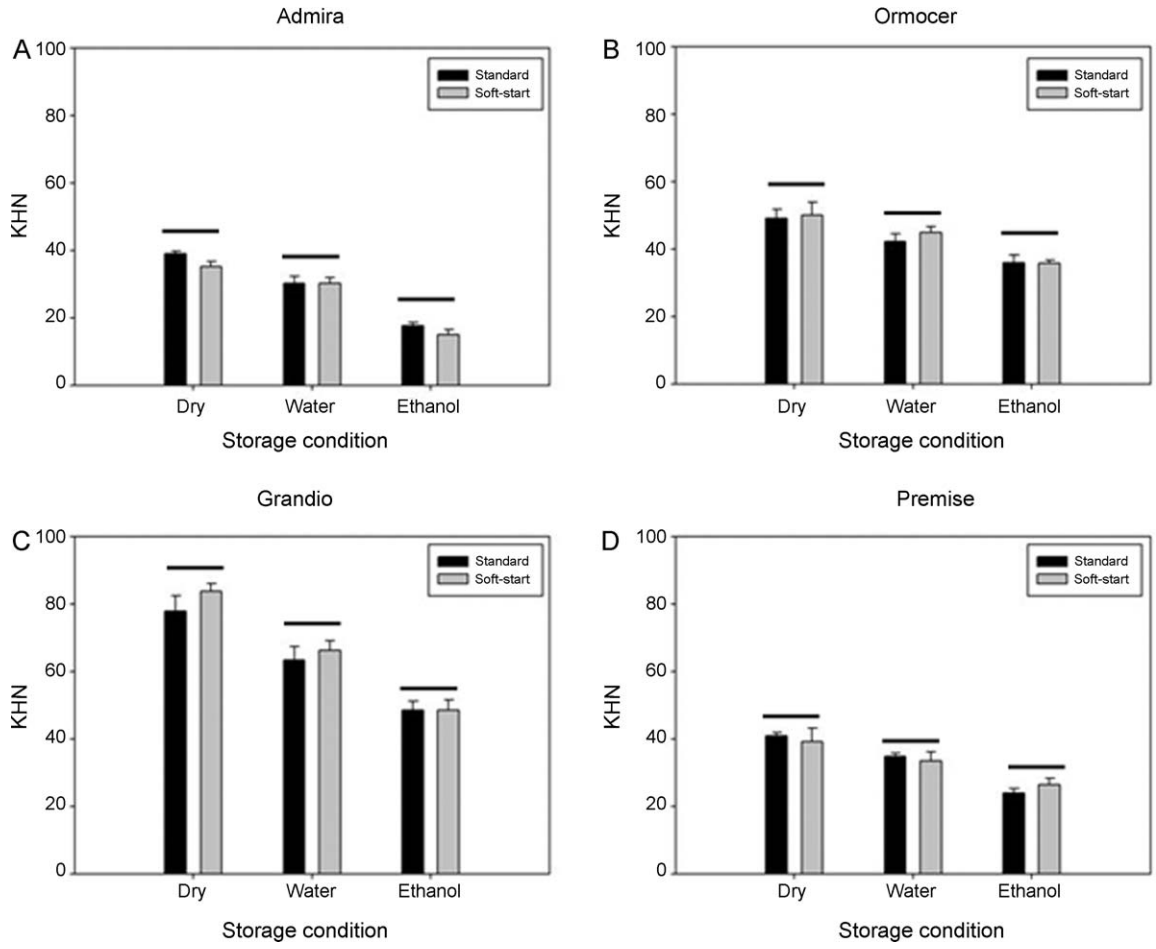

Fig. 2 – Percentage of Knoop hardness change (positive values mean increase in the hardness values, whereas negative values mean decrease), after 7 days storage in different media, for all materials tested submitted to standard and soft-start light curing protocol.

ever, after 1 week in the water, only the experimental Ormocer did not result in reduction of KHN values. The 1 week immersion in ethanol produced a higher deterioration of the surface than the other storage conditions. The experimental Ormocer was the material that exhibited the lowest decrease of the KHN values.

4. Discussion

The material's surface integrity influences the long term clinical performance of resin-composite restorations and such integrity might be affected by several factors. Consequently, the present study investigated the effect of light curing protocols, storage media and materials' formulations on the surface integrity of resin-composite specimens.

Alternative light curing protocols were developed to reduce the inherent polymerization shrinkage stresses created during the application of resin-composite materials [25]. In theory, stress release by viscous flow before the vitrification stage would be allowed to occur without compromising the final polymer properties [26]. Therefore, initial light exposure at lower irradiance values might lead to the formation

Table 2 – Percentage change in hardness (relative to the initial value) of specimens stored in various solvents and photo-polymerized using different curing modes.

Material	Dry and dark		Water		Ethanol	
	Exposure mode		Exposure mode		Exposure mode	
	Continuous	Soft-start	Continuous	Soft-start	Continuous	Soft-start
Experimental ORMOCER	+18%	+19%	+1%	+7%	−14%	−15%
Admira	+26%	+15%	−2%	−2%	−43%	−51%
Grandio	+10%	+23%	−11%	−2%	−31%	−28%
Premise	+8%	+3%	−8%	−12%	−37%	−30%

of a reduced number of polymer growth centres, reducing the reaction rate and decreasing stress development due to the increased opportunity for resin flow before the vitrification stage. Unfortunately, some studies suggest that the final degree of carbon double-bond conversion (DC) tends to be low when using such methods [27]. Furthermore, some other studies suggest that similar DC might be obtained when alternative light curing protocols are used, but the polymeric matrix might exhibit greater softening in solvents due to a possible lower crosslink density (CLD) [14]. In the present study, the Knoop hardness was used as an indirect method to evaluate the DC, and it was observed that, regardless of the material tested, there was no difference of initial KHN values produced by the two light curing protocols tested (Fig. 1). Therefore, the hypothesis that different light curing protocols would affect the initial Knoop hardness (1st hypothesis) was rejected.

As recently mentioned, some researchers suggest that polymers created by different light curing protocols might exhibit similar DC, but distinct CLD. The CLD is an important structural parameter to determine the extent to which the material's surface would be affected by solvent-challenge. The CLD of the studied materials, activated by the two light curing protocols, was assessed through Knoop hardness before and after immersion in different storage conditions. According to the obtained data, both light curing protocols showed no significant difference in CLD, suggesting that the susceptibility to chemical degradation's was not affected by the light curing protocol used, regardless of the material studied and solvent used (Fig. 2). Such outcomes agree with previous researches that demonstrated that the light curing protocol might not affect the CLD [18,22,28]. On the other hand, disagree with studies have shown that different CLD can be produced according to the light curing protocols used [14,15]. The conflicting data can be attributed to differences in light-curing unit, irradiance and VLC mode used. In the present study, the soft-start mode was performed from: 0 up to 1100 mW/cm^2 for 10 s + 1100 mW/cm^2 for 30 s; using an LED unit. The first start of this polymerization process might be enough to produce the majority of the DC, thus there was no time for a differentiation in the polymers chains formation (CLD) compared to the standard mode (1100 mW/cm^2 for 40 s). Therefore, the second hypothesis – that the light curing protocols and the storage conditions would affect the Knoop hardness after 7 days – was partially rejected. Part of the second hypothesis had to be accepted because the storage condition affected the KHN results, regardless of the material tested. The dark storage was used to verify if the post-cure would be affected by the light curing protocol and, as it can be seen, no differences were observed. On the other hand, the ethanol was responsible for the lowest results obtained after 7 days and this ability is probably explained by the solubility parameter, which describes the ability of a molecule to penetrate and dissolve another substance [20].

Regardless of the light curing protocol used (Figs. 1 and 2), the surface integrity of solvent-challenged materials was affected by the materials' formulations and, consequently, the 3rd hypothesis was rejected. Several factors related to the chemistry and structure of the polymer network, are important in determining the extent to which a material will be affected by the oral environment [20]. Important chemical characteristics include the hydrophilicity of the polymer, the difference in the solubility parameter between the polymer and the solvent, and the type of chemical bond within the polymer backbone [12,20,29].

Different performance for each material's category was observed when the samples were immersed in distilled water. While the dimethacrylate-based materials (GRD and PRE) showed a considerable reduction in the KHN values, the Ormocers® (ORM and ADR) had a small decrease or even a slight increase of hardness when stored in water. The inorganic–organic network presented in the ormocers-matrix is different from the organic network used in the dimethacrylate-matrix composites and this can explain our results [30]. Organic–inorganic polymeric hybrid materials are composed of an organic polymer phase and inorganic glasses, or ceramics synthesized by sol–gel processing of organofunctional metal alkoxides [1,31]. The combination of organic and inorganic networks in the ormocers can lead to an alternative dental restorative with reduced polymerization shrinkage and improved resistance to the chemical degradation process [6]. For this purpose, ormocers based on urethane or carboxy-functionalized methacrylate alkoxysilanes [1,2] were introduced into the SiO_2 glass network via the sol–gel approach and were commercially available as an admixed ormocer-matrix, e.g. ADR. The monomer matrix of ADR still contains dimethacrylate monomers [6]. As a result, after curing, unpolymerized dimethacrylate monomers can be eluted. A softening effect was observed when stored in ethanol and/or water for Admira, analogous to that found in conventional dimethacrylate resin-based filling materials.

In an attempt to improve the performance of ADR, a pure ormocer-matrix (ORM) without free dimethacrylate monomers was developed. The pure ormocer-matrix (ORM) was the only material, in this study, that did not show a reduction in the surface hardness values after water storage for 24 h. According to the manufacturer's, the main difference between ADR and ORM is the removal of any methacrylate monomers. This has been shown to reduce water uptake, solubility and volumetric shrinkage [32]. Thus, since water diffuses internally through the resin matrix, filler interfaces, pores and other defects, and then slowly dissolves the filler particles [33], polymers absorb water to different degrees depending on their molecular and microstructural aspects [34]. For this reason, it might be speculated that the modifications made in the pure ormocer-matrix formulation were significantly important to produce a more water stable material compared to the admixed ormocer-matrix and dimethacrylate-matrix composite materials.

In general, for all evaluated materials, the immersion in ethanol induced higher decrease in surface hardness values. As it can be observed, the effect of the solvents in the polymers network was varied. When the forces of attraction between the polymer chains are exceeded by the forces of attraction between solvent molecules and components of the chains [20], the solvent penetrates the resin matrix and disrupts the chains. It is the solubility parameter of the solvent the most important consideration and it determines the extension of the solvent uptake [35,36]. The solvent effect being greatest when there is a minimal solubility parameter mismatch between the solvent and the polymer itself [35,36].

Absolute ethanol has been considered to be among the most aggressive solvents for dental composites network [12,35] as its solubility parameter matches that of the dimethacrylate resin used in composites [37]. However, even in such scenario the pure ormocer-matrix composite (ORM) expressed a noticeable greater resistance to ethanol-challenge than admixed ormocer- (ADR) and dimethacrylate-matrix composites (GRD and PRE), showing that this class of material might be a considerable development for materials with enhanced surface integrity.

5. Conclusions

According to the experimental conditions used in the current study, it is possible to conclude that:

- Different light exposure modes *did not affect* initial Knoop hardness values;
- Light exposure mode did not affect Knoop hardness values after 7 days storage; however the *storage* conditions *did affect* it;
- Surface integrity was affected by the materials' formulations. Compared to the admixed-ormocer and control materials, pure ormocer-matrix preserved surface integrity after water storage and had the greatest resistance to the ethanol-challenged.

Acknowledgment

The authors would like to acknowledge VOCO Dental Company and Kerr for kindly providing their materials.

REFERENCES

[1] Wolter H, Storch W, Ott H. New inorganic/organic copolymers (ORMOCERs) for dental applications. Mater Res Soc Symp Proc 1994;346:143–9.
[2] Wolter H, Storch W, Ott H. Novel carboxy functionalized sol–gel precursors. Mater Res Soc Symp Proc 1996;435:67–71.
[3] Stanley HR. Local and systemic responses to dental composites and glass ionomers. Adv Dent Res 1992;6:55–64.
[4] Meryon SD, Riches DW. A comparison of the in vitro cytotoxicity of four restorative materials assessed by changes in enzyme levels in two cell types. J Biomed Mater Res 1982;16:519–28.
[5] Polydorou O, Trittler R, Hellwig E, Kummerer K. Elution of monomers from two conventional dental composite materials. Dent Mater 2007;23:1535–41.
[6] Moszner N, Gianasmidis A, Klapdohr S, Fischer UK, Rheinberger V. Sol–gel materials. 2. Light-curing dental composites based on ormocers of cross-linking alkoxysilane methacrylates and further nano-components. Dent Mater 2007:26.
[7] Tagtekin DA, Yanikoglu FC, Bozkurt FO, Kologlu B, Sur H. Selected characteristics of an Ormocer and a conventional hybrid resin composite. Dent Mater 2004;20:487–97.
[8] Janda R, Roulet JF, Latta M, Ruttermann S. Water sorption and solubility of contemporary resin-based filling materials. J Biomed Mater Res B: Appl Biomater 2007;82:545–51.
[9] Cavalcante LM, Masouras K, Watts DC, Pimenta LA, Silikas N. Effect of nanofillers' size on surface properties after toothbrush abrasion. Am J Dent 2009;22:60–4.
[10] Chung SM, Yap AU. Effects of surface finish on indentation modulus and hardness of dental composite restoratives. Dent Mater 2005;21:1008–16.
[11] Shahdad SA, McCabe JF, Bull S, Rusby S, Wassell RW. Hardness measured with traditional Vickers and Martens hardness methods. Dent Mater 2007;23:1079–85.
[12] Schneider LF, Moraes RR, Cavalcante LM, Sinhoreti MA, Correr-Sobrinho L, Consani S. Cross-link density evaluation through softening tests: effect of ethanol concentration. Dent Mater 2007.
[13] Ferracane JL. Correlation between hardness and degree of conversion during the setting reaction of unfilled dental restorative resins. Dent Mater 1985;1:11–4.
[14] Asmussen E, Peutzfeldt A. Influence of pulse-delay curing on softening of polymer structures. J Dent Res 2001;80:1570–3.
[15] Asmussen E, Peutzfeldt A. Two-step curing: influence on conversion and softening of a dental polymer. Dent Mater 2003;19:466–70.
[16] Asmussen E, Peutzfeldt A. Influence of selected components on crosslink density in polymer structures. Eur J Oral Sci 2001;109:282–5.
[17] Asmussen E, Peutzfeldt A. Polymer structure of a light-cured resin composite in relation to distance from the surface. Eur J Oral Sci 2003;111:277–9.
[18] Witzel MF, Calheiros FC, Goncalves F, Kawano Y, Braga RR. Influence of photoactivation method on conversion, mechanical properties, degradation in ethanol and contraction stress of resin-based materials. J Dent 2005;33:773–9.
[19] Yap AU, Soh MS, Han TT, Siow KS. Influence of curing lights and modes on cross-link density of dental composites. Oper Dent 2004;29:410–5.
[20] Ferracane JL. Hygroscopic and hydrolytic effects in dental polymer networks. Dent Mater 2006;22:211–22.
[21] Soh MS, Yap AU. Influence of curing modes on crosslink density in polymer structures. J Dent 2004;32:321–6.
[22] Pfeifer CS, Braga RR, Ferracane JL. Pulse-delay curing: influence of initial irradiance and delay time on shrinkage stress and microhardness of restorative composites. Oper Dent 2006;31:610–5.
[23] Moon HJ, Lee YK, Lim BS, Kim CW. Effects of various light curing methods on the leachability of uncured substances and hardness of a composite resin. J Oral Rehabil 2004;31:258–64.
[24] Aguiar FH, Braceiro AT, Ambrosano GM, Lovadino JR. Hardness and diametral tensile strength of a hybrid composite resin polymerized with different modes and immersed in ethanol or distilled water media. Dent Mater 2005;21:1098–103.
[25] Bouschlicher MR, Rueggeberg FA, Boyer DB. Effect of stepped light intensity on polymerization force and conversion in a photoactivated composite. J Esthetic Dent 2000;12:23–32.
[26] Lim BS, Ferracane JL, Sakaguchi RL, Condon JR. Reduction of polymerization contraction stress for dental composites by two-step light-activation. Dent Mater 2002;18:436–44.
[27] Lu H, Stansbury JW, Bowman CN. Impact of curing protocol on conversion and shrinkage stress. J Dent Res 2005;84:822–6.
[28] Emami N, Soderholm KJ. Dynamic mechanical thermal analysis of two light-cured dental composites. Dent Mater 2005;21:977–83.
[29] Bagheri R, Tyas MJ, Burrow MF. Subsurface degradation of resin-based composites. Dent Mater 2007;23:944–51.
[30] Yap AU, Lim LY, Yang TY, Ali A, Chung SM. Influence of dietary solvents on strength of nanofill and ormocer composites. Oper Dent 2005;30:129–33.

[31] Manhart J, Kunzelmann KH, Chen HY, Hickel R. Mechanical properties of new composite restorative materials. J Biomed Mater Res 2000;53:353–61.
[32] Neumann T. Physical Properties of a New ORMOCER®-Based Experimental Filling Material IADR-PEF, Dublin 2006;59.
[33] Kalachandra S, Wilson TW. Water sorption and mechanical properties of light-cured proprietary composite tooth restorative materials. Biomaterials 1992;13:105–9.
[34] Mortier E, Gerdolle DA, Dahoun A, Panighi MM. Influence of initial water content on the subsequent water sorption and solubility behavior in restorative polymers. Am J Dent 2005;18:177–81.
[35] Wu W, McKinney JE. Influence of chemicals on wear of dental composites. J Dent Res 1982;61:1180–3.
[36] McKinney JE, Wu W. Chemical softening and wear of dental composites. J Dent Res 1985;64:1326–31.
[37] Kao EC. Influence of food-simulating solvents on resin composites and glass-ionomer restorative cement. Dent Mater 1989;5:201–8.