Influence of pre-cure temperature on Vickers microhardness of resin composite

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ABSTRACT

Objectives: The aim was to assess the effects of the pre-heating or pre-cooling resin composite on surface hardness.
Methods: Sixty test specimens (TS) were made of resin composite Z250, using a steel matrix 3mm x 5mm, light cured with an XL3000 light cure appliance. The TS were divided into 6 groups of 10 specimens, altering the light cure time (CT), 20 or 40s, and the resin composite temperature (TEMP), 5°C, 24°C or 54°C. The TS were stored in distilled water at 37°C for 24 hours and the Vickers microhardness analysis was performed. Both the TS top and bottom were analyzed.
Results: The results of two-way ANOVA showed that the temperature had a significant effect on microhardness. At the top, higher microhardness values were observed for the temperature of 54°C, while the temperature of 5°C and 24°C presented no significant differences.
Conclusion: It was concluded that pre-heating resin composite significantly increased the resin microhardness, the light cure time of 40 s improved top microhardness for the use of resin at 24 °C or 5 °C.

Key-words: composite resins, temperature, microhardness

Introduction

Restorative resin composites (RC) have gained an outstanding role in modern Dentistry as a result of the growing demand for esthetic restorations. On the other hand, these materials still do not have ideal physical properties and are frequently inadequately manipulated, so that limited advantage is taken of their qualities.

A big problem associated with their use is insufficient cure, due to the application of light sources of inadequate intensity(1-3), use of shorter cure time than is necessary, or due to the distance of the light tip from the material to be activated.(4)

A correlation has been reported between surface microhardness and degree of conversion(5) such that the former (e.g. Vickers hardness number, VHN) may be appropriate as a simple monitor of mechanical strength.

A comparison of top and bottom surface hardness of disc specimens, where the activation curing tip is in contact with the top surface only, may be taken as an indicator of the "depth-of-cure"(6), and it is suggested that the bottom/top ratio should be in excess of 0.8 for adequate cure.(7)

It is known that the cure process is no more than a sequence of chemical chain reactions, in which double bonds from the carbon link are broken in order for the molecules to bond, forming the polymer, resulting in an exothermic reaction. Furthermore, it is known that the higher the temperature, the shorter the period of induction, which is one in which the initiator molecules become energized or activated and start transferring energy to the monomer molecules.(8) Some studies have proved that the use of resin composites at temperatures higher than ambient temperature promote a greater immediate conversion of the monomers, while exposed to the light source, reducing the post-cure that occurs in the absence of light.(9-12)

With the objective of better conservation of the material, many manufacturers recommend storing resin composites under refrigeration. If such materials are used at this temperature, one could suppose that retardation of the reaction speed could occur, resulting in a cure beyond the ideal.

Based on this, the purpose of this study was to compare post-cure top and bottom microhardness number (VHN), post cure bottom surface/top surface VHN ratios of a commercial resin composite material exposed and cured at a nominal temperature of 5°C, 24°C and 54°C.

The null hypothesis are that:

- There is no difference in post-cure mean surface microhardness for discs of RC (top and bottom surface, respectively) that have been pre-heated or pre-cooling and light cured for different times (20 or 40s);
- There is no difference in post-cure mean surface microhardness ratios (bottom/top) for discs of RC

(top and bottom surface, respectively) that have been pre-heated or pre-cooling and light cured for different times (20 or 40s);

Materials and method

To make the test specimens, the resin composite Z250 (3M, St. Paul, MN, USA) shade A2 (lot 1MN), was inserted in a single increment in a steel matrix 3 mm deep x 5 mm in diameter. A polyester strip was placed over the material, and a glass slide was pressed down on top of it to obtain a flat surface. The glass slide was then removed and the tip of the light cure appliance XL3000 (3M, St. Paul, MN, USA) with energy density of 550mW/cm^2 was placed directly onto the polyester strip, touching it.

Sixty specimens were made and divided into 6 groups of 10 specimens, altering the light polymerization time and the resin composite temperature. All the specimens were performed in a room with a temperature controlled at 24°C.

To make the test specimens at 5^{o}C, the syringe of resin composite was submerged in water at 5^{o}C taking care not to contaminate the material with water, the temperature being gauged by means of a bulb thermometer. The syringe remained in this bath for a minimum of 15 minutes before making the first test specimen, in order to guarantee that all the material was at the same temperature, as found in the pilot study. After each test specimen was made, the syringe of resin was again submerged in the water at the desired temperature. This temperature was selected because it is the temperature usually used in refrigerators to conserve products.

To make the test specimens with resin at 54^{o}C, the well sealed syringe of resin composite was submerged in water at 54^{o}C, together with the bulb thermometer. After each test specimen was made, the syringe of resin was again submerged in the water at the desired temperature. The water was kept at 54^{o}C by means of a histological cut bath receptacle, which allowed heating to be controlled.

The test specimens were stored in distilled water at 37^{o}C for 24 hours, in dark flasks that prevented the passage of light, in order to allow delayed polymerization and water absorption. Next, the Vickers microhardness analysis was performed, by means of a micro hardness tester FM-700 (Future Tech Corp., Tokyo, Japan) using a 50g load with a dwell time of 10s. Both the test specimen top and bottom surfaces were analyzed. For each side, three indentations were made in order to calculate a mean value.

To analyze the effects of temperature and polymerization time on resin composite microhardness, as well as the interaction between these factors, two-way analysis of variance (ANOVA) was performed, independently for the top and for the bottom. To detect which groups showed significant differences among them, the Tukey test was used. To detect the presence of significant differences between the top and the bottom of specimens for each group, the non paired t test was performed. The microhardness ratios (bottom/top) was also calculated for each group. For all statistical analysis, a level of significance of 5% was adopted.

Results

The results of the two-way ANOVA for the top and bottom of specimens can be observed in Table 1.

There was a statistically significant difference between the measurements taken at the top and the bottom for some groups (Table 2).

The t test demonstrated that there are significant differences in microhardness between the top and bottom of specimens, for all the groups, exhibiting significantly higher means for the top.

In Table 1 it can be seen that the temperature had a significant effect on microhardness, both at the top and bottom of specimens. Table 2 shows that at the top, higher microhardness values were observed for the temperature of 54^{o}C, while the temperature of 5 and 24^{o}C presented no significant differences.

With regard to light activation time, Table 1 shows that it had a significant influence on microhardness only at the bottom of specimens, demonstrating a trend towards a higher microhardness value for the time of 40 s (Table 2).

It was found that the factor temperature interacted significantly with the factor time, with regard to increase in microhardness (Table 1).

In Table 2, it can be observed that only the 5^{o}C/40 s group obtained a bottom/top ratio of 0.84. In accordance with the specifications of the International Organization for Standardization (ISO) number 4049:2000(13,14) the bottom must have at least 80% of the top microhardness for polymerization to be considered efficient.

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FACTORS	TOP		BOTTOM	
	F	P	F	p
Temperature	54.86	0.00*	10.80	**0.00***
Time	0.61	0.43	21.06	**0.00***
Temperature x Time	7.37	0.00*	3.59	**0.03***

***** Significant differences***

Table 1 - Results of two-way ANOVA for the top and bottom of specimens

VHN	Top	Bottom	Bottom/top ratio
Group #1: 5°C/20s	75.24(3.49) [a*]	52.44 (5.49) [a]	**0.70**
Group #2: 5°C/40s	73.07(2.55) [a]	61.67 (6.25) [a,b]	**0.84**
Group #3: 24°C/20s	69.82(1.70) [a]	45.00 (6.10) [c]	**0.64**
Group #4: 24°C/40s	74.19(3.90) [a]	58.60 (7.39) [a]	**0.78**
Group #5: 54°C/20s	81.14 (2.71) [b]	61.03 (6.06) [a,b]	**0.75**
Group #6: 54°C/40s	80.64(1.66) [b]	62.97 (9.53) [b]	**0.80**

***** Similar superscripts indicate no significant difference (p<0.05)***

Table 2 – Mean (SD) surface microhardness and bottom/top ratio.

Discussion

The degree of resin composite conversion can be assessed by various methods, included microhardness, being associated with the light intensity of the light curing appliances, type of light curing appliance, cure time, post-cure time and resin composite depth(15) .

In the present study, another parameter that could alter the top microhardness of resin composite could be observed, which was a change in its pre-cure temperature. Heating to 54°C significantly increased the top microhardness, irrespective of the cure time (20 or 40s). This could mean a greater conversion of the resin composite, induced by the heat, as was observed by various authors(9,10,16,6,11), which could reduce the degree of degradation of the restoration over the course of time. This is because it has been established that the lower the degree of cure of a given solid polymer, the lower would be its resistance(8).

According to Yap et al(3), the effectiveness of cure generally increases with increase in exposure to light. This occurs because there is greater conversion of the resinous monomers. With the increase in temperature, the initial conversion of monomers is accelerated and post-cure is significantly reduced(9-11). According to Trujillo et al.(11), the gain in post-cure conversion is observed in cure performed at ambient temperature, whereas with heating the resin composite to 54.5°C there was no significant post-cure conversion. According to Friedman(17), heating resin composite to 130°F (54.5°C) propitiates a more complete cure, and depending on the composite used, an increase of 8 to 17% in the degree of cure could occur, and the light-cure time could be reduced by 50 to 80%. This could explain the results of the present study, in which it was observed that the top microhardness did not vary when using the light-cure time of 20 or 40s, that is to say, the maximum degree of cure of the specimen top was attained in half the light-cure time.

According to Friedman(17), heating the resin composite to 130°F produces some advantages. In addition to reducing the cure time by 50 to 80%, the resin presents a reduction in its viscosity, which could facilitate its adaptation in the cavity by flowing, as it has a consistency similar to that of honey. Heating must be done using compules that can be syringed to make them applicable for clinical use.

With regard to the possibility of heating the resin composite to 130°F being able to affect the pulp tissue, according to Friedman(17), insertion of the composite heated to this temperature only increases the pulp temperature by 1.6 °C if there is 1mm of remaining dentin, which would cause no damage. However, the authors believe that this technique is not clinically recommended, before further studies are conducted to prove the small amount of pulp heating, as according to Zach and Cohen(18), the pulp temperature cannot be increased by more than 5.5°C in order to maintain its vitality. Furthermore, according to Loney and Price(19), the thickness of the remaining dentin interferes in the increase in pulp temperature, and the temperature that the resin composite attains when some Halogen light curing appliances are used, could affect the pulp integrity. For Hussey et al(20), who also observed that the increase in the resin composite temperature during its light curing could interfere in pulp vitality, indicate the practice of pulp protection and placement of a base under the restoration.

Another interesting observation is that when the resin composite is light cured more quickly, there may be greater shrinkage which could cause an increase in marginal gaps and consequently more microleakage. In this context, various studies have been conducted to seek a gradual resin composite polymerization, in which light curing is started with a lower light intensity, which is then increased up to the maximum intensity that the light curing appliance can produce(21). Thus, it allows resin composite flow during polymerization, and consequently less stresses produced, reducing marginal gap formation, with the consequent reduction in marginal microleakage. Some studies, however, found no reduction in marginal gap formation and shrinkage when using the gradual polymerization technique(22-24,3). In this context, it becomes necessary for studied to be conducted to prove whether there is any alteration in microleakage when pre-heated resin is used.

Littlejohn et al(9) assessed the percentage of conversion of resin composite at various temperatures, among them 5 and 25°C and observed that there was a greater degree of conversion at the temperature of 25°C, and the higher the resin temperature was before its polymerization, the higher the degree of conversion. In the present study, there were no significant alterations with regard to the microhardness of resin composite Z250 when it was inserted in the matrix at ambient temperature (24°C) or cooled (5°C), which is in contrast to the above-mentioned study. This could mean that resin composite stored in a refrigerator and immediately inserted in the cavity for restoration would have no relation to microhardness.

In the present study, the specimen bottom presented a lower degree of conversion. In accordance with ISO 4049:2000 the resin composite bottom is satisfactorily polymerized when it attains at least 80% of the top polymerization. In Table 2, it can be observed that only the 5°C/40 s group obtained a bottom/top ratio of 0.84. It could be concluded that the resin Z250 must not be used in 3 mm increments even if the light cure time is increased to 40 s.

Conclusions

The results obtained in this study according to the proposed methodology were as follows:
- Heating the resin composite Z250 significantly increased the top microhardness.
- Ambient temperature (24°C) or cooling (5°C) the resin composite did not interfere significantly in the microhardness of the top or the bottom.
- The polymerization time of 40 s improved the top microhardness for the use of resin at ambient temperature (24°C) or cooled (5°C).
- When the resin composite was heated to 54°C the polymerization time did not influence the microhardness.

CONFLICTS OF INTEREST

The authors declare that they have no conflicts of interest.

References

1. Barghi N, Berry T, Hatton C. Evaluating intensity output of curing lights in private dental offices. J Am Dent Assoc 1994; 125:992-996.

2. Abate PF, Zahra VN, Macchi RL. Effect of photopolymerization variables on composite hardness. J Prosthet Dent 2001; 86:632-635.

3. Yap AU, Ng SC, Siow KS. Soft-start polymerization: influence on effectiveness of cure and post-gel shrinkage. Oper Dent 2001; 26:260-266.

4. Hansen EK, Asmussen E. Visible-light curing units: correlation between depth of cure and distance between exit window and resin surface. Acta Odontol Scand 1997; 55:162-166.

5. DeWald JP, Ferracane JL. A comparison of four modes of evaluating depth of cure of light-activated composites. J Dent Res 1987; 66:727-730.

6. Awliya WY. The influence of temperature on the efficacy of polymerization of composite resin. J Contemp Dent Pract 2007; 8:9-16.

7. Pilo R, Cardash HS. Post-irradiation polymerization of different anterior and posterior visible light-activated resin composites. Dent Mater 1992; 8:299-304.

8. Phillips R (1991) Skinner's science of dental materials. 9th edn. Saunders, Philadelphia.

9. Littlejohn L (2003) Curing efficiency of a direct composite at different temperatures. Paper presented at the 32nd Annual Meeting and Exhibition of the AADR, San Antonio, March 12-15.

10. Vogel K, Rueggeberg F, Rheinberger V (2003) Influence of initiator system and temperature on real time double bond conversion. Paper presented at the

32nd Annual Meeting and Exhibition of the AADR, San Antonio, March 12-15.

11. Trujillo M, Newman SM, Stansbury JW. Use of near-IR to monitor the influence of external heating on dental composite photopolymerization. Dent Mater 2004; 20:766-777.

12. Lucey S, Lynch CD, Ray NJ, Burke FM, Hannigan A. Effect of pre-heating on the viscosity and microhardness of a resin composite. J Oral Rehabil 37:278-282.

13. 4049 ISI (2000) Polymer based filling, restorative and luting materials vol ISO 4049, 3rd edn.,

14. Dunn WJ, Bush AC. A comparison of polymerization by light-emitting diode and halogen-based light-curing units. J Am Dent Assoc 2002; 133:335-341.

15. Price RB, Derand T, Loney RW, Andreou P. Effect of light source and specimen thickness on the surface hardness of resin composite. Am J Dent 2002; 15:47-53.

16. Munoz CA, Bond PR, Sy-Munoz J, Tan D, Peterson J. Effect of pre-heating on depth of cure and surface hardness of light-polymerized resin composites. Am J Dent 2008; 21:215-222.

17. Friedman J. Thermally assisted flow and polymerization of composite resins. Contemporary Esthetic Restorative Practice 2003; 2:46.

18. Zach L, Cohen G. Pulp Response to Externally Applied Heat. Oral Surg Oral Med Oral Pathol 1965; 19:515-530.

19. Loney RW, Price RB. Temperature transmission of high-output light-curing units through dentin. Oper Dent 2001; 26:516-520.

20. Hussey DL, Biagioni PA, Lamey PJ. Thermographic measurement of temperature change during resin composite polymerization in vivo. J Dent 1995; 23:267-271.

21. Mehl A, Hickel R, Kunzelmann KH. Physical properties and gap formation of light-cured composites with and without 'softstart-polymerization'. J Dent 1997; 25:321-330.

22. Amaral CM, Castro AK, Pimenta LA, Ambrosano GM. [Effect of techniques of composite resin insertion and polymerization on microleakage and microhardness]. Pesqui Odontol Bras 2002; 16:257-262.

23. Sahafi A, Peutzfeldt A, Asmussen E. Soft-start polymerization and marginal gap formation in vitro. Am J Dent 2001; 14:145-147.

24. Tarle Z, Meniga A, Ristic M, Sutalo J, Pichler G, Davidson CL. The effect of the photopolymerization method on the quality of composite resin samples. J Oral Rehabil 1998; 25:436-442.

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