

How does duration of curing affect the radiopacity of dental materials?

Arash Poorsattar Bejeh Mir, Morvarid Poorsattar Bejeh Mir*

Dentistry Student Research Committee (DSRC), Dental Materials Research Center, Dentistry School, Babol University of Medical Sciences, Babol, Iran

*Private Practice of Orthodontics, Montreal, Quebec, Canada

ABSTRACT

Purpose : Clinicians commonly encounter cases in which it is difficult to determine whether adjacent radiopacities are normal or pathologic. The ideal radiopacity of composite resin is equal to or higher than that of the same thickness of aluminum. We aimed to investigate the possible effects of different curing times on the post-24-hour radiopacity of composite resins on digital radiographs.

Materials and Methods : One mm thick samples of Filtek P60 and Clearfil resin composites were prepared and cured with three regimens of continuous 400 mW/cm² irradiance for 10, 20 and 30 seconds. Along with a 12-step aluminum step wedge, digital radiographs were captured and the radiopacities were transformed to the equivalent aluminum thicknesses. Data were compared by a general linear model and repeated-measures of ANOVA.

Results : Overall, the calculated equivalent aluminum thicknesses of composite resins were increased significantly by doubling and tripling the curing times ($F(2,8)=8.94$, $p=0.002$). Notably, Bonferroni post-hoc tests confirmed that the radiopacity of the cured Filtek P60 was significantly higher at 30 seconds compared with 10 seconds ($p=0.04$). Although the higher radiopacity was observed by increasing the time, other comparisons showed no statistical significance ($p > 0.05$).

Conclusion : These results supported the hypothesis that the radiopacity of resin composites might be related to the duration of light curing. In addition to the current standards for radiopacity of digital images, defining a standard protocol for curing of dental materials should be considered, and it is suggested that they should be added to the current requirements for dental material. (*Imaging Sci Dent 2012; 42 : 89-93*)

KEY WORDS : Radiography, Dental, Digital; Composite Resins; Light-Curing of Dental Adhesives

Introduction

In recent decades, there have been many attempts to improve the physical, chemical, and mechanical properties of restorative resin composites in terms of wearing resistance, stability, and esthetics. Mechanical properties are related to the type and quality of structural monomers, degree of conversion, silanization, initiators, and size of fillers.¹

Shrinkage of resin composites during and after curing

is concern to clinicians and manufacturers since it may result in restoration-tooth gap with further cusp fracture, tooth sensitivity, or recurrent caries. Volumetric shrinkage is an indicator of the degree of conversion. The smaller the filler particles and the higher the monomer viscosity, the lower the degree of conversion and polymerization shrinkage.^{1,2}

According to the American Dental Association (ADA) recommendation, adequate radiopacity is one of requisites of dental materials.³ Proper radiopacity aids a dentist to distinguish a restorative material from the adjacent decalcified dentin and secondary caries, and to localize voids or gingival overhangs.⁴ Preferably, restorative materials should have a radiopacity higher than that of enamel.⁵ Although this simple comparative reference has many advantages,

Received March 8, 2012; Revised March 30, 2012; Accepted April 19, 2012
Correspondence to : Dr. Arash Poorsattar Bejeh Mir
Dentistry School, Babol University of Medical Sciences, Ganj Afroz Ave, Babol, Mazandaran Province, Iran
Tel) 98-151-321-6383, Fax) 98-151-321-6383, E-mail) arashpoorsattar@yahoo.com

How does duration of curing affect the radiopacity of dental materials?

specifying a set of various thicknesses of enamel and dentin and the equivalent thicknesses of aluminum for each would expand its generalizability.⁴

Various methods have been introduced for assessing the radiopacity of dental materials, including digitalized and digital radiography, absorbance densitometry, and spectrophotometry. Based on ADA/ANSI recommendations, there were studies which validated the method of reporting the equivalent aluminum radiopacity of restorative materials.^{4,6} According to the last declaration of ISO4049:2009, “if the manufacturer claims that the material is radiopaque, the radiopacity shall be equal to or greater than that of the same thickness of aluminum and no less than 0.5 mm below any value claimed by the manufacturer”.⁷ Moreover, an X-ray source performing within the range of 65 ± 5 kVp using D-speed and No. 4 sized film should be applied.⁷ Aluminum step wedge may be produced from either a single block or stacking 1 mm-thick strips of aluminum.^{6,7} The material should have at least 98% purity of aluminum containing less than 0.1% copper and less than 1% iron. Accordingly, the measured equivalent radiopacity of dental materials is influenced by the purity of the aluminum to which it is

compared.⁷

The final arrangement of the polymer matrix of a composite resin affects its molecular density and consequently, its radiopacity. This may be related to the degree of conversion and shrinkage of resin composites.⁶ The aim of the present study was to assess the possible effect of different curing times on the radiopacity of restorative composite resins.

Materials and Methods

Sample Preparation

One-mm-thick samples of Filtek P60 (3M/ESPE, St. Paul, MN, USA) and Clearfil (Kuraray, Tokyo, Japan) composite resins were prepared (i.e., 15 samples of each resin composite with A₂ shade). The samples were processed using a brass-made 15 mm × 15 mm × 1 mm mold. Photo-activation was performed with a quartz-tungsten-halogen (QTC) source by three protocols with a continuous irradiation of 400 mW/cm² intensity. The first five rectangular samples were irradiated for 10 seconds at each corner pole and the

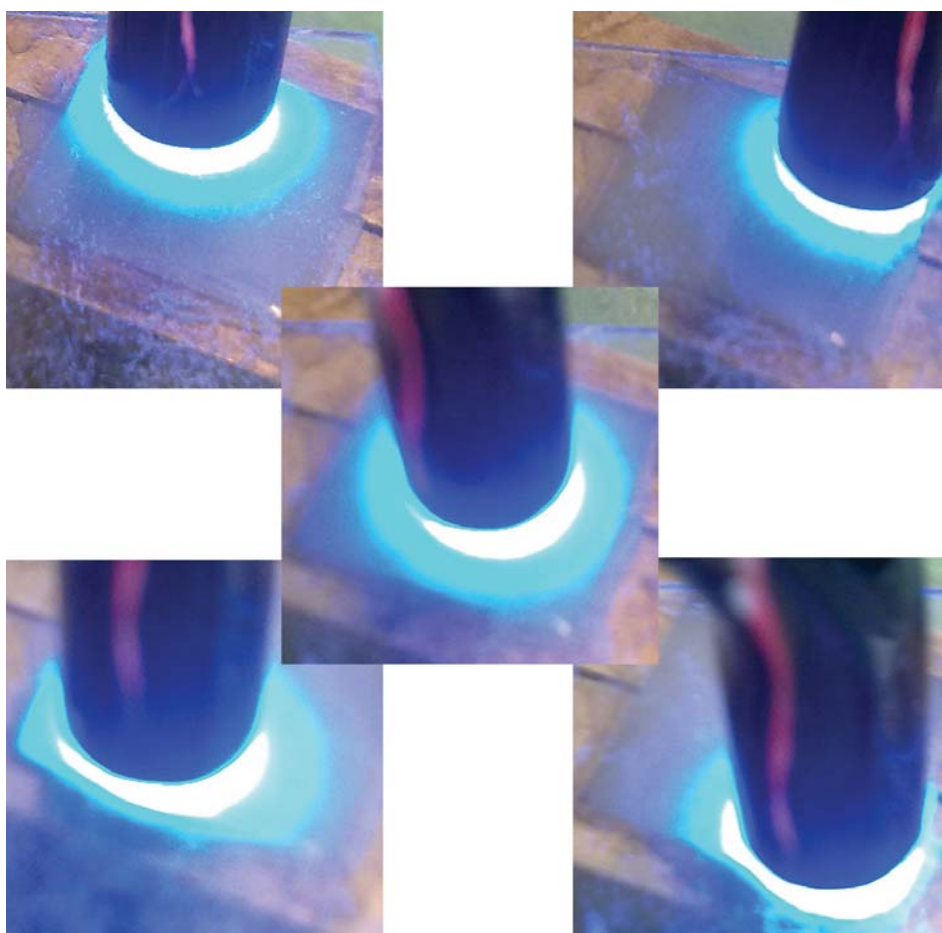


Fig. 1. Each sample is irradiated in the five different poles: upper right, lower right, lower left and upper left plus the central zone.

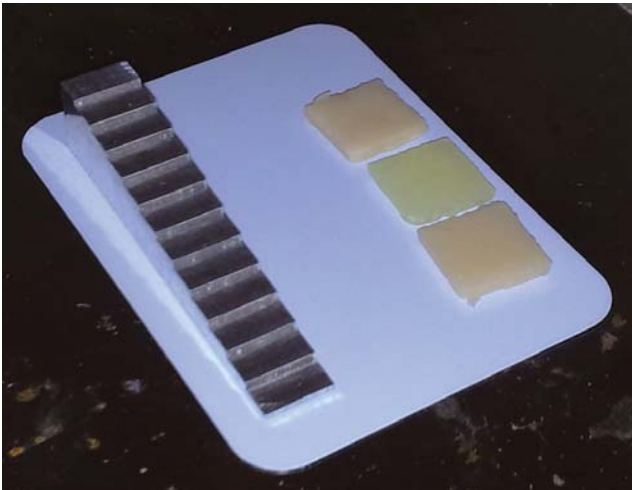


Fig. 2. The random sets of prepared samples are arranged on an occlusal photostimulable phosphor (PSP) receptor along with a 12-step aluminum step wedge.

center of each sample (overall, five zones for each sample) (Fig. 1). Consecutive sets (each consisting of five samples) were cured by doubling and tripling the duration for each area with the same intensity. Deficient samples with large apparent voids in radiography were replaced by sound ones (Fig. 2).

Radiopacity Calculation

Various series (each composed from random selection of three samples from a total of 30 prepared and cured samples) were radiographed along with 12-step aluminum (93% purity, Radravesh Shomal Co., Babolsar, Iran) (Fig. 2). All radiographic images were taken using a dental X-ray unit (Minray, Soredex, Helsinki, Finland) and a photostimulable phosphor (PSP) plate (Digora, Soredex, Helsinki, Finland) with the following setup: 70 kVp, focus-object distance of 30 cm, and 0.2 seconds exposure time. Radiopacity was measured using Digora for Windows (ver. 2.5, Digora, Soredex, Helsinki, Finland) software. Radiopacity was measured in five different areas, each with a 40×40 pixel area (i.e., four corner poles plus the central zone) in order to reduce the measurement bias. The mean calculated radiopacity of each sample was then taken into account. Thereafter, the mean radiopacity was transformed to the equivalent aluminum thickness with the equation obtained from plotting the aluminum thickness against the correspondent radiopacity of the step wedge exposed with the same setup. The reliability was checked by re-measuring 7 random samples two weeks later and an acceptable Pearson's correlation ($r=0.85$) was obtained. In addition, all of the

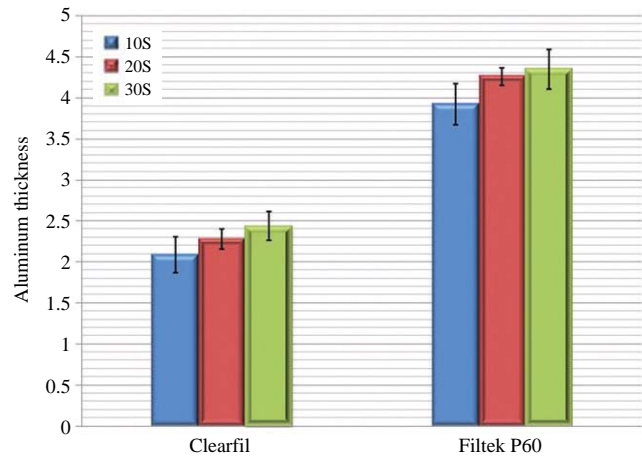


Fig. 3. Varying trends of absorbencies of Filtek P60 and Clearfil with different curing regimens (10, 20, and 30 seconds curing time).

measures were performed by a single operator to eliminate inter-observer inconsistency. The intra-observer agreement scored at $R^2=0.56$, which was acceptable.

Statistics

Tracing the varying trends of radiopacity was performed by applying a general linear model (GLM) and repeated-measures of ANOVA. Sphericity (one of the ANOVA assumptions) was tested with Mauchly's test. Homogeneity of variances was tested with Levene's statistic. Moreover, in order to have a more precise analysis of paired groups, post-hoc multiple comparisons and Bonferroni test were performed.

Results

The final radiopacity and equivalent thickness of single-, double-, and triple-cured samples are displayed in Figure 3. The calculated changes of both materials were statistically significant ($F(2)=8.94$, $p=0.002$, Estimated power 0.93). A significant incrementally increasing trend was observed with increased duration of light curing for Filtek P60 ($F(2,8)=6.13$, $p=0.02$, estimated power=0.73). The radiopacity of Clearfil increased with increased curing time, however the changes showed no significant level ($F(2,8)=3.25$, $p=0.092$, estimated power=0.45). Overall, a statistically significant increase was observed for the comparison of 10 seconds to 30 seconds of curing, meanwhile marginally significant ($p=0.059$) and no significant changes ($p=0.681$) were found for 10 to 20 seconds and 20 to 30 seconds comparisons, respectively. Multiple comparisons revealed a significant increase from 10 to 30 seconds light

curing regimen for Filtek P60 ($p=0.04$), while the other comparisons were not significant (Filtek P60: 10 seconds vs. 20 seconds $p=0.19$, 20 seconds vs. 30 seconds $p=1$; Clearfil: 10 seconds vs. 20 seconds $p=0.59$, 10 seconds vs. 30 seconds $p=0.27$, 20 seconds vs. 30 seconds $p=0.85$). A larger effect was related to the differences between 10 and 20 seconds of light curing time.

Discussion

In the present study, the impact of different light curing regimens, in terms of duration of curing, on the radiopacity of composite resins was assessed. It was determined that doubling and tripling the curing time would considerably increase the radiopacity that was captured and processed by a digital radiography. To the knowledge of the authors, this might be the first study that evaluated the effect of light-curing time on the radiopacity of resin composites on digital radiographic images. The present study did not follow two rules of the ADA. First, the purity of the step wedge was 93%. The calculated R^2 of the regression model for predicting aluminum density by step wedge thickness was 0.923. We had no access to the standard 98% pure step wedge. The low-pure step wedge, however, would be against the current recommendation which would inevitably decrease the reliability of the method, comparing the R^2 of 0.923 with the R^2 of as high as 0.98-0.99 when a standard step wedge was used.^{6,7} More precise comparisons might be achieved when a standard step wedge and calibrated units with the same energy output would be applied.⁸ Contrary to the method of the step wedge, which might be biased by the percentage purity of the step wedge, a method of calculating the attenuation coefficient that was not widely influenced by purity was proposed more recently.⁹ As for the second issue, we used a digital X-ray system. As mentioned above, the ADA dictated D-speed film and not a digital sensor. There have been no guidelines or standards for such a digital system. A further confirmatory study would be advisable to compare conventional and digital receptors in more depth.

With the passage of light through uncured resin, the ion viscosity is first reduce and thereafter reaches a plateau toward a relatively acute slope.¹⁰ The ultimate ion viscosity is an indicator of the degree of conversion and final hardness. Hardness may be evaluated to qualify the polymerization.¹¹ A variety of variables such as light intensity, sample thickness, exposure time, and temperature may control the kinetics of polymerization.¹⁰ We used 1 mm thick samples. Within this limit, it was expected that the

upper and lower surface of the resin composite had approximately the same degree of conversion. We assumed that the average calculated radiopacity of two surfaces might not significantly differ though the data were not shown. A difference would be especially obvious if thick samples were irradiated briefly. We evaluated the radiopacity 24 hours after light curing due to the conclusion of previous studies that polymerization slowly progressed from 16 minutes to 24 hours. It should be mentioned that there was no consensus on the degree of post-exposure polymerization over time.¹² Initiators are activated by light curing and subsequently polymerization progresses as free radicals are released and the double carbon-carbon bond changes to single bonds. Hence, the 0.3-0.4 nm van der Waals molecular distance is replaced by 0.15 nm covalence bonds and a 15% volumetric shrinkage occurs.^{2,13} Therefore, such a tighter molecular rearrangement with increased density might explain the increased radiopacity when doubling and tripling the curing time.

It seems that different systems and setups might be responsible for these disagreements. Nevertheless, current radiopacifiers (e.g., zirconia) are responsible for the radiopacity of dental material that is irrelevant of corresponding thickness.⁶ Notably, this may hamper the differences achieved by different curing protocols. With a given shade, changes in radiopacity are more related to the size of the monomer particles. As the particle size is reduced, with a parallel to higher disturbance rate of light energy, absorption decreases. Smaller particles have a wider interaction surface and lower mobility, therefore the degree of conversion and volumetric changes are reduced.¹⁴ Presumably, different short and intense, weak and long, or soft star regimens may differentially change the radiopacity with regards to the different polymerization rates.¹⁵

The present study had several limitations. First, the results of tracing the actual polymer matrix changes during polymerization and curing were indeed different from the laboratory findings. These results might be originated from our ignoring the confounding effect of the configuration factors and elastic behaviour of composite resins, which indeed moderated the matrix configuration and molecular arrangement during light curing.¹ In addition, the bonding effect of the adhesives was not quantified as another confounder. Furthermore, in the present in-vitro assessment of composite resin radiopacity, the attenuating effect of intraoral hard and soft tissue on the measured radiopacity was not considered. Finally, while curing deep cavities using incremental techniques, inevitably more than 1 mm would exist between the tips of the light curing device and

the bottom layer of the composites. This mishap would attenuate the degree of conversion and possibly influence the trend of radiopacity change.

Future investigations considering different curing regimens, different particle sizes of the same shade (e.g., comparing hybrid- and nano-sized) with various configuration factors and post-polymerization assessment time would be recommended. Interestingly, volumetric changes and perhaps radiopacity alteration during light curing may be moderated by replacing traditional Bis-GMA monomers by Silorane (combination of siloxane + oxirane). Oxirane rings are extended during polymerization; hence they compensate the shrinkages to some extent.¹⁶ Although minimum radiopacity requirements were well documented, maximum limits have not been clarified considering some commercially available filling materials with extreme radiopacity (e.g., as high as the equivalent of 11 mm of aluminum). Such a high radiopacity may outperform the range of measurement of digital radiographs of limited latitude with 256 scales with consequent difficulty in interpretation of radiographs.¹⁷ It seems that ADA/ANSI/ISO4049 recommendations have missed the attention to dictate the standards of light curing. Besides, the hard and soft tissue attenuation effects ought to be added to previously recommended guidelines. A better recommendation may also include special considerations for various cavity preparations.

In conclusion, the light curing time affected the radiopacity of composite resins on the radiographs. Accordingly, these results should be considered when the radiopacity of composite resins would be reported for informative purposes and comparative decisions. Also, the standards for light curing of resin composites should be incorporated into the current guidelines.

References

- Zimmerli B, Strub M, Jeger F, Stadler O, Lussi A. Composite materials: composition, properties and clinical applications. A literature review. *Schweiz Monatsschr Zahnmed* 2010; 120 : 972-86.
- Nagem Filho H, Nagem HD, Francisconi PA, Franco EB, Mondelli RF, Coutinho KQ. Volumetric polymerization shrinkage of contemporary composite resins. *J Appl Oral Sci* 2007; 15 : 448-52.
- American Dental Association. Obstacles to the development of a standard for posterior composite resins. Council on Dental Materials, Instruments, and Equipment. *J Am Dent Assoc* 1989; 118 : 649-51.
- Imperiano MT, Khoury HJ, Pontual ML, Montes MA, Silveira MM. Comparative radiopacity of four low-viscosity composites. *Braz J Oral Sci* 2007; 6 : 1278-82.
- Cook WD. An investigation of the radiopacity of composite restorative materials. *Aust Dent J* 1981; 26 : 105-12.
- Gu S, Rasimick BJ, Deutsch AS, Musikant BL. Radiopacity of dental materials using a digital X-ray system. *Dent Mater* 2006; 22 : 765-70.
- International Organization for Standardization. ISO 4049: 2009. Dentistry - Polymer based restorative materials. 4th ed. Geneva: ISO; 2009.
- Watts DC, McCabe JF. Aluminium radiopacity standards for dentistry: an international survey. *J Dent* 1999; 27 : 73-8.
- Nomoto R, Mishima A, Kobayashi K, McCabe JF, Darvell BW, Watts DC, et al. Quantitative determination of radiopacity: equivalence of digital and film X-ray systems. *Dent Mater* 2008; 24 : 141-7.
- Malhotra N, Mala K. Light-curing considerations for resin-based composite materials: a review. Part II. *Compend Contin Educ Dent* 2010; 31 : 584-91.
- Jeong TS, Kang HS, Kim SK, Kim S, Kim HI, Kwon YH. The effect of resin shades on microhardness, polymerization shrinkage, and color change of dental composite resins. *Dent Mater J* 2009; 28 : 438-45.
- Leung RL, Fan PL, Johnston WM. Post-irradiation polymerization of visible light-activated composite resins. *J Dent Res* 1983; 62 : 363-5.
- Pereira RA, Araujo PA, Castañeda-Espinosa JC, Mondelli RF. Comparative analysis of the shrinkage stress of composite resins. *J Appl Oral Sci* 2008; 16 : 30-4.
- Halvorson RH, Erickson RL, Davidson CL. The effect of filler and silane content on conversion of resin-based composite. *Dent Mater* 2003; 19 : 327-33.
- Price RB, Felix CA, Andreou P. Evaluation of a second-generation LED curing light. *J Can Dent Assoc* 2003; 69 : 666.
- Weinmann W, Thalacker C, Guggenberger R. Siloranes in dental composites. *Dent Mater* 2005; 21 : 68-74.
- Heo MS, Choi DH, Benavides E, Huh KH, Yi WJ, Lee SS, et al. Effect of bit depth and kVp of digital radiography for detection of subtle differences. *Oral Surg Oral Med Oral Pathol Oral Radiol Endod* 2009; 108 : 278-83.