EGTRIB Journal
JOURNAL OF
THE EGYPTIAN SOCIETY OF TRIBOLOGY
VOLUME 13, NO. 2, April 2016, pp. 25 - 37 **ISSN 2090 - 5882**

EFFECT OF CURING PROCESS OF DENTAL NANOCOMPOSITE RESIN ON SHORE HARDNESS

Meshref A. A., Mazen A. A., El-Giushi M. A. and Ali W. Y.

Production Engineering and Design Department, Faculty of Engineering, Minia University, P. N. 61111, El-Minia, EGYPT.

ABSTRACT
The objectives of this study is to determine the effect of light curing modes and curing times on the Shore hardness of hybrid composite resin filled with titanium dioxide (TiO_2) nanoparticle.The materials used were hybrid composite resin and titanium dioxide nanoparticles (TiO_2 nanoparticles). Four groups of composite resin specimens were prepared; one as received and three groups reinforced with TiO_2 nanoparticles in concentrations of 0.1, 0.2 and 0.3 wt. %. TiO_2 nanoparticles were manually mixed with hybrid composites resin. The resulting paste was packed into plastic bars. The specimens were then polymerized from both sides for 10, 20, 40, 60 and 80 seconds using a visible light curing unit. The light polymerization modes used were gradually strong, flashing and strong mode. Surface hardness was measured using Shore D durometer test. The use of gradually strong curing mode resulted in lower hardness for the unreinforced hybrid composites resin filled with TiO_2 nanoparticles compared with the hardness obtained using the flashing mode and strong curing mode. Specimens of curing time 10, 20 and 40 seconds exhibited lower hardness compared to 60 and 80 seconds curing time. This study indicated that, strong curing mode produced dental nanocomposite resin with higher Shore hardness, where 80 seconds curing time gave higher values of Shore hardness. Shore hardness results are significantly affected by curing times compared to curing modes.

KEYWORDS
Titanium dioxide, nanoparticles, hybrid composite resin, dental nanocomposite, shore hardness, polymerization modes, curing times.

INTRODUCTION
Composite resin is an accepted alternative for many restorations in the rearward teeth. Composite resin has changed considerably since their introduction in the early 1960s. Improvements in mechanical and physical properties have allowed composite resin to play a progressively important role in dental restorative materials, [1]. The mechanical properties of dental restorative materials are extremely important for their clinical execution and continuation. Dental restorative materials need to resist the high occlusal

forces that occur in the mouth during chewing and to be resistant to ensure steady curative success. These forces can reach the values of 200 N in the frontal occlusal area, and up to 800 N in the lateral occlusal segments, or even up to 3500 N during some abnormal jaw movements and teeth contacts, the mechanical properties of dental composite resin can be affected by various factors; these factors are shown in Fig. 1, [2].

So as to improve the mechanical properties such as hardness, compressive strength and fracture toughness of composite resin, earlier studies have concentrated on the evolution of curing techniques and pretreatment of inorganic fillers and resin monomers, [3 - 4]. Nowadays, light curing dental materials are extensively used in the field of dentistry. A sufficient polymerization of composite resin is essential for the extreme success of the dental restorations. The degree of cure of composite resin materials influences their mechanical properties, biocompatibility, dimensional stability, solubility and color change, [5]. The subject of polymerization in the field of dentistry has become very argumentative, particularly in regard to high intensity against low intensity light sources. High intensity light sources are attractive to the dentist, because they can basically reduce polymerization time, [1]. The most common light sources used in dentistry to polymerize dental composite resin are blue light emitting diode (LED) and quartz tungsten halogen, [6]. In the year 2000, 94% of U.S. dentists used visible light curing units for curing dental composite resin which used as restorative materials for both anterior and posterior restorations, [7]. The LED light curing unit is vastly used and displays good results in polymerization of the dental composite resin. In addition, the LED light source showed a homogeneous curing across the surface of the dental composite resin and good values of Vickers hardness, [8].

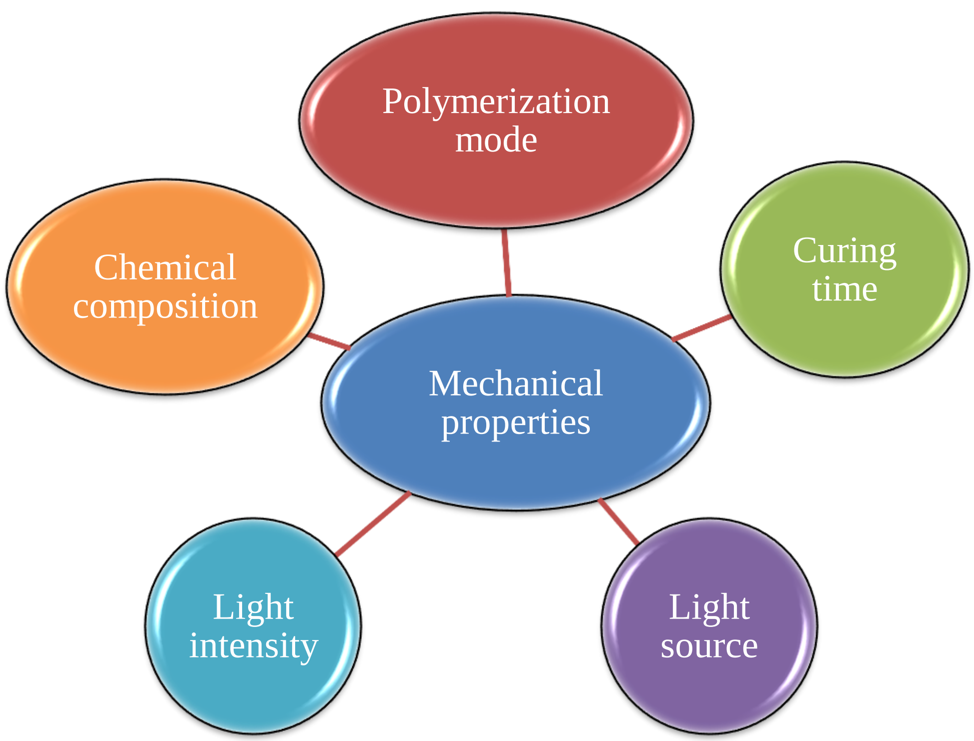


Fig. 1 Factors influence mechanical properties of dental composite resin.

Different light curing modes are obtainable such as soft start, step curing, or oscillating irradiation. These special polymerization modes have been considered to increase the degree of conversion for improving the properties of the material, and to decrease internal stress to obtain better marginal quality in bonded dental composite resin, [9 - 10]. The mechanical properties of the dental composite resin are related to the quality of polymerization process. The polymerization depth of the dental composite resins depends on the polymerization time, the light intensity and the amount of visible light transmitted through the material, [8]. Polymerization time depends on several factors, such as light intensity, resin thickness, resin shade, curing through tooth structure, composite filling and box deep, [11]. Various combinations of light intensity and curing time can lead to important differences in the properties of the material within certain energy density. Dentists have an important role in the quality of their restoration by the suitable choice of curing conditions i.e. curing modes and curing times, [12].

The use of nanoparticles has become an important area of research in dentistry field. Titanium dioxide nanoparticles (TiO_2 nanoparticles) have been suggested for use as reinforcing fillers to dental composite resin and epoxy. TiO_2 as an inorganic filler has many promising properties as it is non-toxic, biocompatible and chemically stable, [13]. TiO_2 nanoparticles also have premium mechanical properties, for example, the modulus of elasticity is approximately 230 GPa, and it is inexpensive with titanium being the fourth most abundant metal on earth, following aluminum, iron and magnesium, [14]. In a recent investigation, TiO_2 nanoparticles reinforced dental composite resin were found to have improved flexural strength and microhardness, [15].

One of the most used indirect procedures to estimate the degree of curing of the dental composite resin is the hardness test, [8]. In the present paper, Shore durometer hardness was used to evaluate the hardness of the tested specimens. The durometer is a hand sized instrument that measures the indentation hardness of rubber and plastic products. Durometer hardness is the resistance of the material being tested to the penetration of the indenter as a result of a variable force applied to the indenter by a spring, [16]. Several studies have examined the influence of light curing modes and polymerization times on the mechanical properties of dental composite resin. Aljosa et al., [2], found that the influence of the light curing mode on mechanical properties is material and property dependent. The soft start polymerization mode mostly produced higher hardness values of all tested dental materials. On the other hand, Farias et al., [17] concluded that the different light curing modes do not have an important effect on the mechanical properties of the composites used in the study.

The aim of the present work is to determine the effect of light curing modes (gradually strong, flashing and strong modes) and curing times 10, 20, 40, 60 and 80 seconds on the Shore hardness of hybrid composite resin filled with titanium dioxide (TiO_2) nanoparticle.

EXPERIMENTAL

The materials used were a commercially available hybrid composite resin of shade A1 and titanium dioxide nanoparticles (TiO_2 nanoparticles) with diameter of < 25 nm. Detailed information about the materials used in the present work is shown in Tables 1 and 2.

Table 1 Details of Composite Resin.

Description	Classification	Manufacturer	Shade	Curing	Lot No.
Visible light cure, Resin-based dental restorative material	Hybrid composite	Prime-Dent, U.S.A.	A1	Light cure	YL08Q

Table 2 Details of TiO_2 Naoparticles.

Diameter	Density	Surface area
21 nm	4 g cm^{-3}	50 m^2 g^{-1}

PREPARATION OF TEST SPECIMENS

Four experimental groups of hybrid composite resin specimens were prepared; three groups containing TiO_2 nanoparticles in different concentrations of 0.1, 0.2 and 0.3 wt. %, and one in as-received condition, contained no TiO_2 nanoparticles. TiO_2 nanoparticles were weighed using a balance of accuracy of 0.001 g and added to the hybrid composites resin. TiO_2 nanoparticles and the hybrid composites resin were mixed by hand on a mixing paper. Before curing, the resulting paste was packed into plastic bars 6 mm in diameter and 10 mm in height. The specimens were then polymerized from both sides using a visible light curing unit (LED) for 10, 20, 40, 60 and 80 seconds. After curing, the specimens were removed from the bars and ground with grinding paper (1000 grit size) and then polished. Fig. 2 shows method of preparation of specimens. Fifteen specimens were fabricated for each group using gradually strong curing mode, flashing curing mode and strong curing mode. The dimensions and shape of specimen are shown in Figures 3 and 4, respectively.

POLYMERIZATION OF SPECIMENS

The light source used in this study to polymerize composites is blue light emitting diode (LED). Figure 5 shows the polymerization of the specimens by light emitting diode source. Detailed information about the LED is shown in the Table 3. The light polymerization modes used were as follows; gradually strong, flashing and strong mode. Gradually strong polymerization mode means emitting of a reduced light energy at the beginning of polymerization, and increase the energy gradually until it reaches full intensity at the end of polymerization (Fig.6 (a)). Flashing polymerization mode means

emitting of full light energy and then disappears (Fig. 6 (b)). Strong polymerization mode means emitting of a constant, stable, full-intensity light-energy (Fig. 6 (c)).

Table 3. Details of the Light Emitting Diode (LED)

Intensity (mW/cm^2)	Spectral emission (nm)	Time and depth
1200 - 2000	420 - 480	5s/3 mm

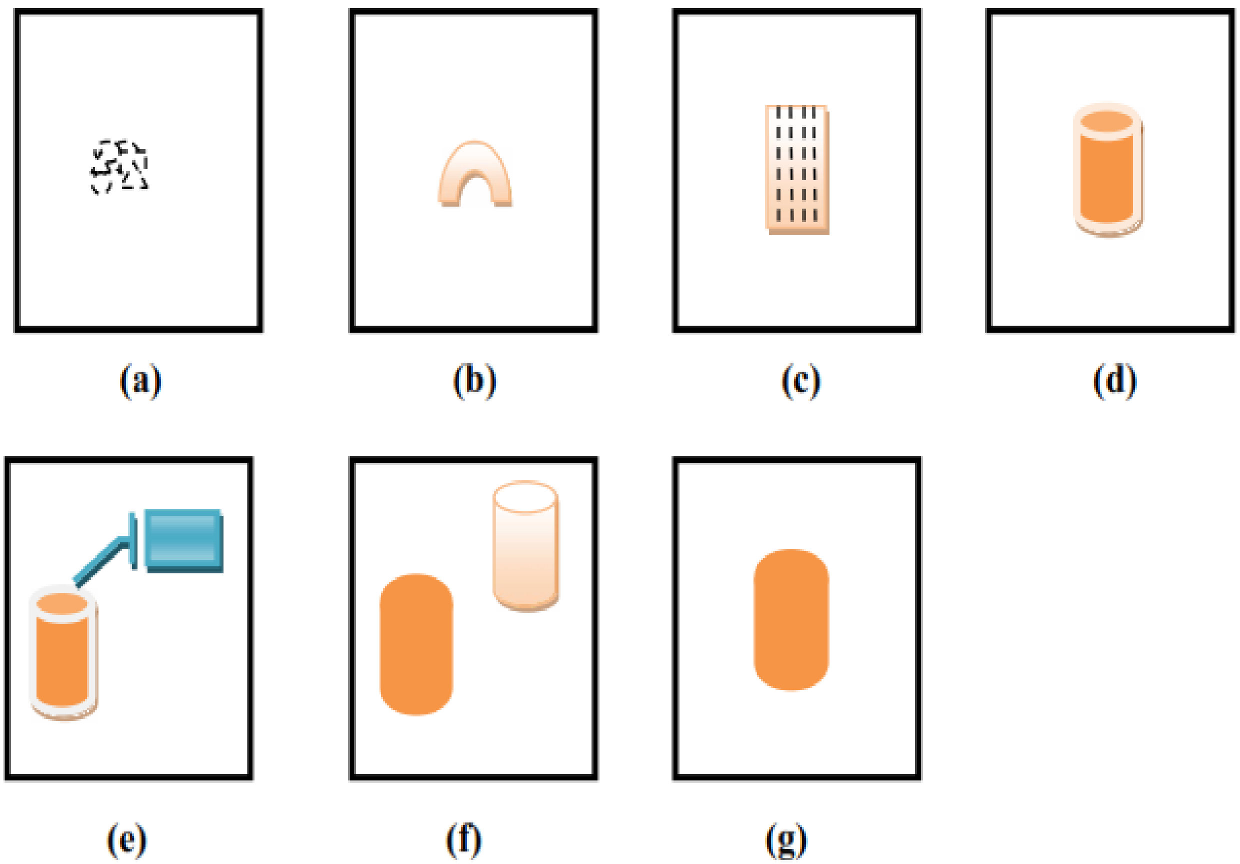


Fig. 2 Method of preparation of specimen (a) TiO_2 nanoparticles (b) Composite resin (c) Mixing (d) Packing (e) Curing (f) Removing (g) Grinding and final shape of specimen.

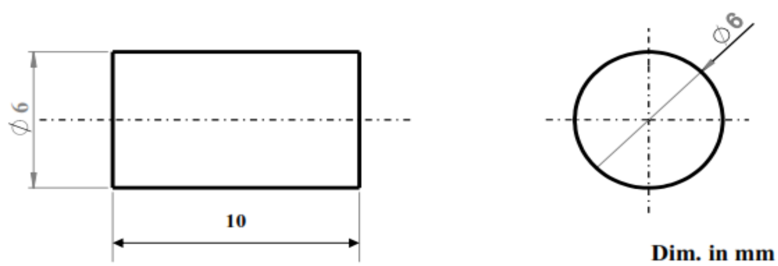


Fig. 3 Dimensions of specimen.

Fig. 4 Shape of specimen.

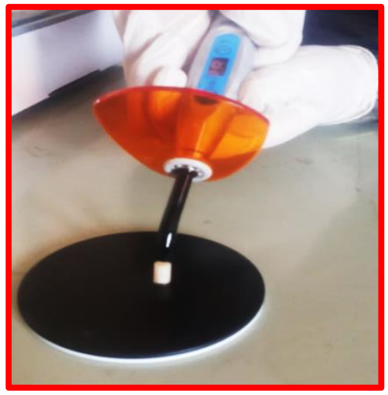
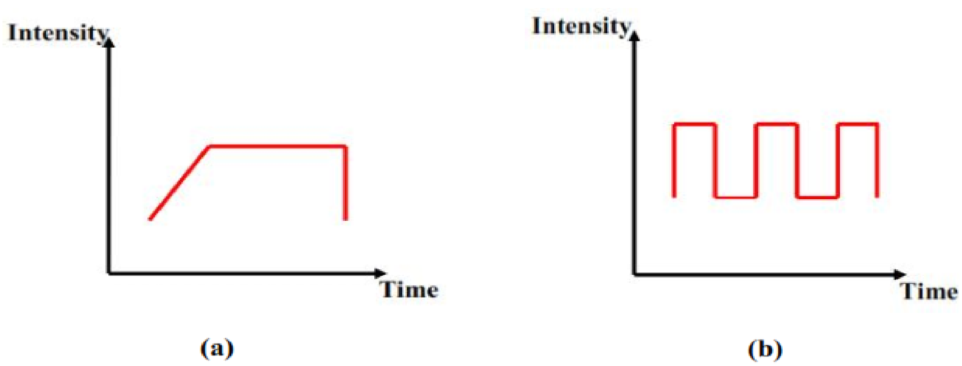


Fig. 5 Polymerization of the specimens by light emitting diode source.



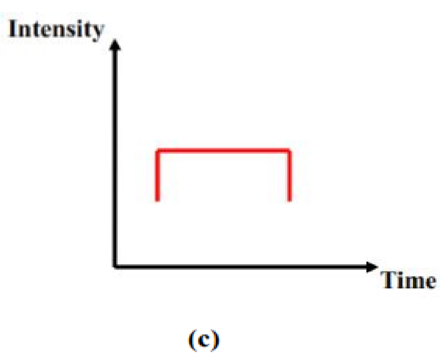


Fig. 6 Light-curing modes (a) gradually strong mode (b) flashing mode (c) strong mode.

To measure the surface hardness of the specimens, cylindrical specimens (diameter = 6 mm, height = 10 mm) were prepared for each group. Shore D durometer instrument was used with dial value (1-100 degree). Hardness was measured on both sides of the specimens (top and bottom). Four measurements per specimen were carried out, two on top and two on bottom side of each specimen. The average of the four measurements was considered as the hardness of the specimen.

RESULTS AND DISCUSSION

Results obtained by Shore hardness test are shown in Figures 7 – 13. For each polymerization time and concentration of TiO_2 nanoparticles hardness was measured on top and bottom side of the specimens. Shore hardness of the gradually strong mode, flashing mode and strong mode are shown in Fig.7 for as received specimens. Shore hardness of the three modes increased as the polymerization time increased. From Fig.7, it is clear that flashing mode and strong mode showed higher shore hardness compared to gradually strong mode. Shore hardness of flashing mode increased by 6 % at polymerization time 40 seconds compared to gradually strong mode. Hardness results of specimens polymerized by flashing mode are similar to these polymerized by strong mode.

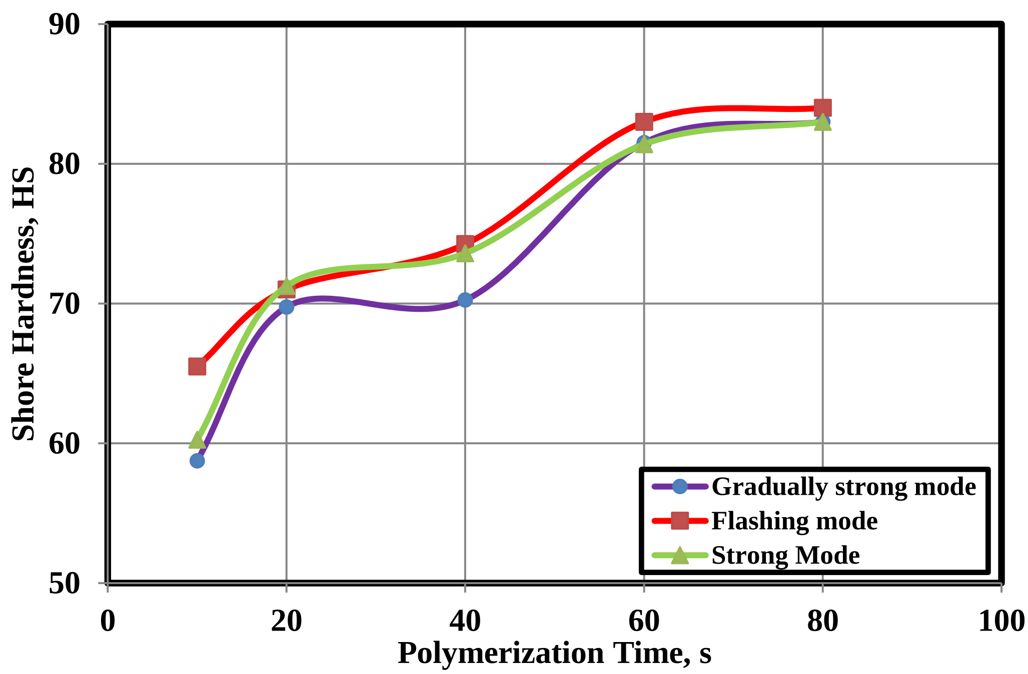


Fig. 7 Relationship between hardness and time for as received specimens.

For 0.1 wt. % TiO_2 nanoparticles composites, Shore hardness of the three modes increased as the polymerization time increased. Strong mode showed higher shore hardness compared to flashing mode and gradually strong mode. Gradually strong curing mode produced dental nanocomposite resin with lower hardness. From Fig. 8, it

can be seen that strong curing mode may be recommended as the best technique for curing the dental nanocomposite resin.

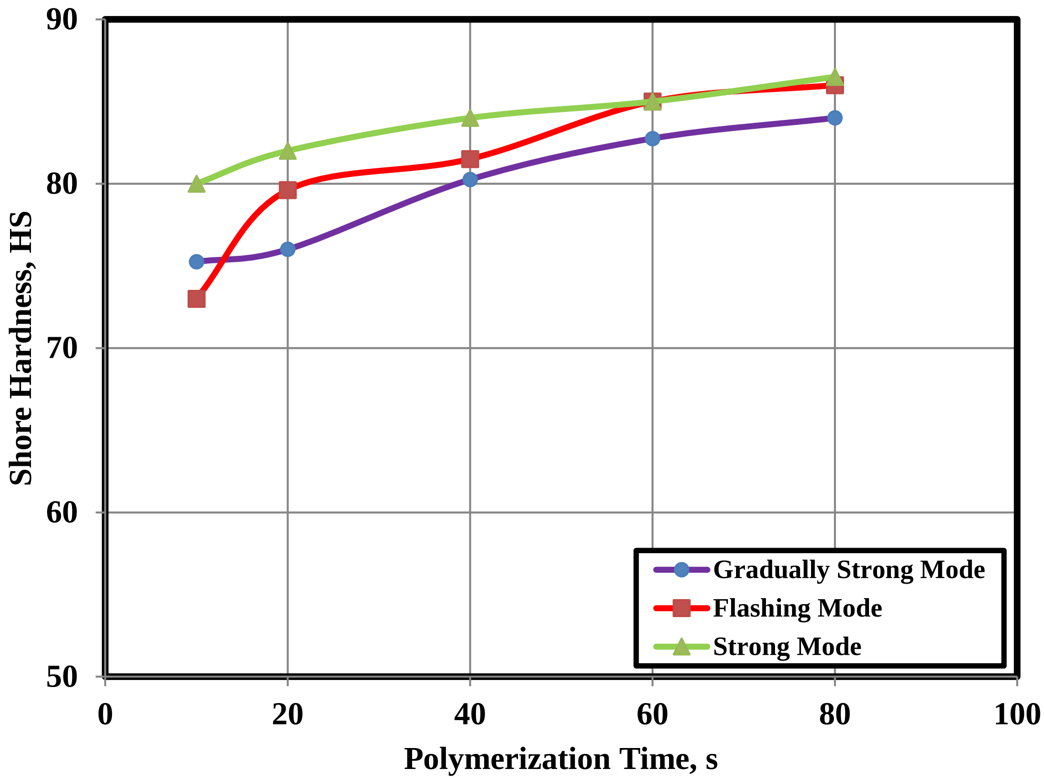


Fig. 8 Relationship between hardness and time for 0.1 wt. % TiO_2 nanoparticles group.

Figure 9 shows Shore hardness of the gradually strong mode, flashing mode and strong mode for 0.2 wt. % TiO_2 nanoparticles group. As shown from Fig. 9, Shore hardness of the three modes increased with the increase in the polymerization time. Strong mode showed higher shore hardness compared to flashing mode and gradually strong mode up to 60 seconds polymerization time. An increase of 5 % in Shore hardness of strong mode is observed at polymerization time 20 seconds compared to gradually strong mode. Hardness results of specimens polymerized by strong curing mode are similar to these polymerized by flashing curing mode at polymerization time 60 and 80 seconds.

Shore hardness of the gradually strong mode, flashing mode and strong mode for 0.3 wt. % TiO_2 nanoparticles group are shown in Fig. 10. Shore hardness of the three modes increased with the increase in the polymerization time. From Fig. 10, it is clear that strong mode showed higher shore hardness compared to flashing mode and gradually strong mode. Gradually strong curing mode produced dental nanocomposite resin with lower hardness.

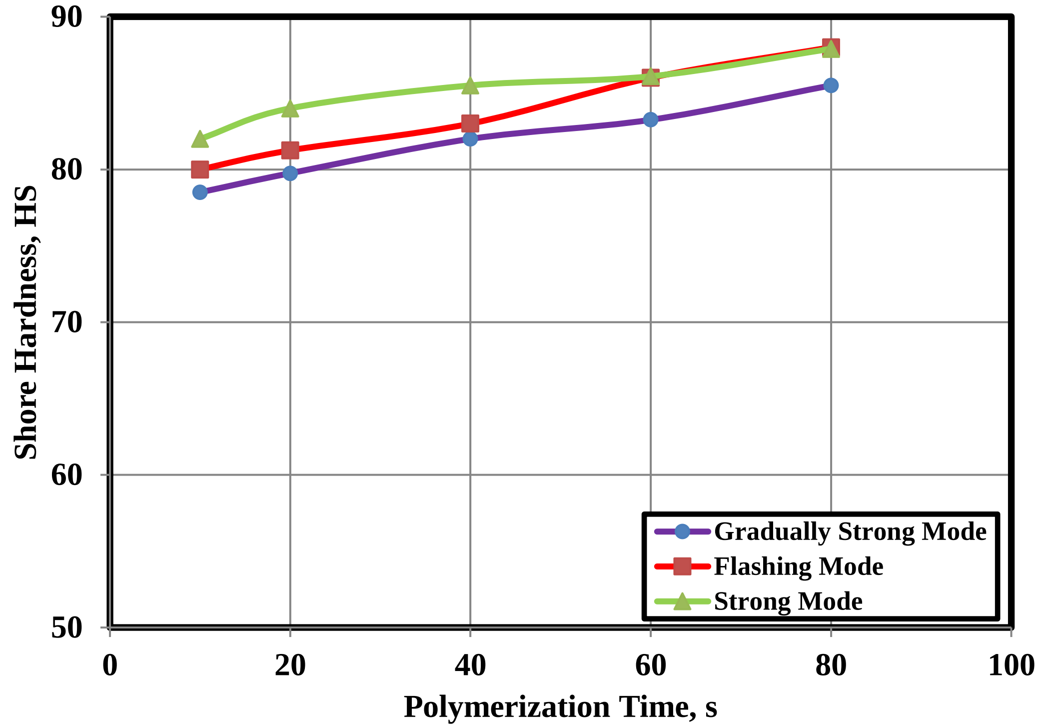


Fig. 9 Relationship between hardness and time for 0.2 wt. % TiO_2 nanoparticles group.

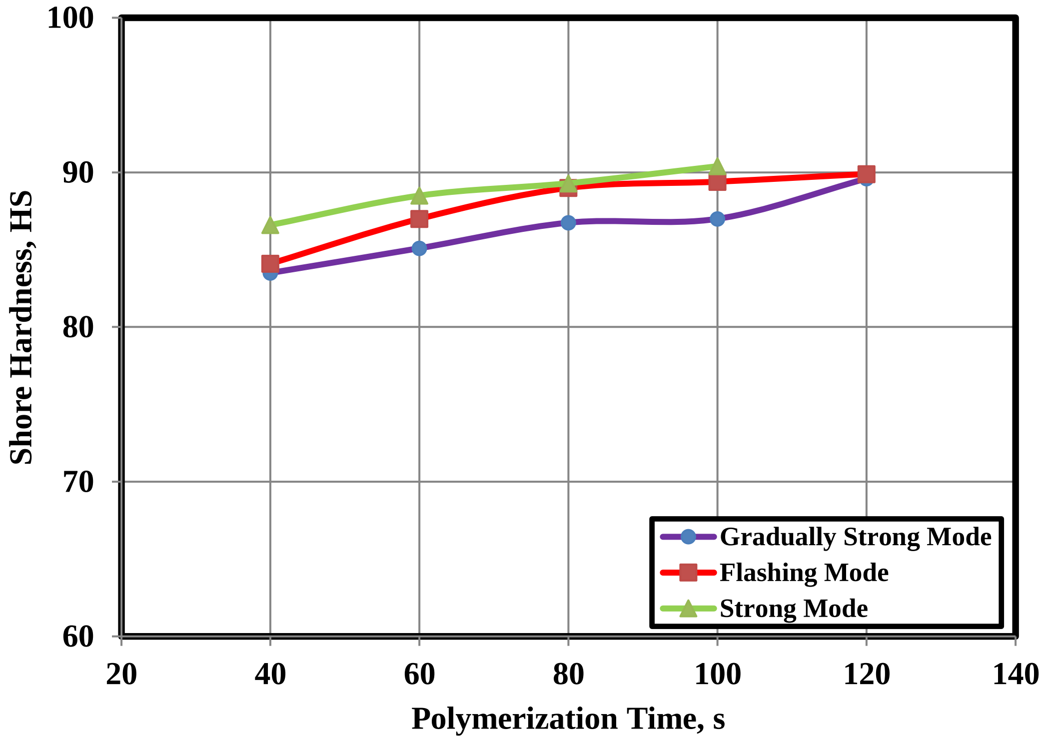


Fig. 10 Relationship between hardness and time for 0.3 wt. % TiO_2 nanoparticles group.

Shore hardness of hybrid composite resin and nanocomposite resin at 10, 20, 40, 60 and 80 seconds curing time are plotted as a function of the concentration of TiO_2 nanoparticles in Fig. 11 It is clear that Shore hardness of the tested materials increases with increasing curing time and the concentration of TiO_2 nanoparticles. The obtained results as shown in Fig. 11, revealed that curing times showed pronounced effects on Shore hardness at all used curing times. Shore hardness of curing time 80 seconds increased by 12 % at 0.1 wt. % TiO_2 nanoparticles compared to curing time 10 seconds. The highest Shore hardness values for the material used in this work were observed in 80 seconds curing time followed by 60 seconds curing time.

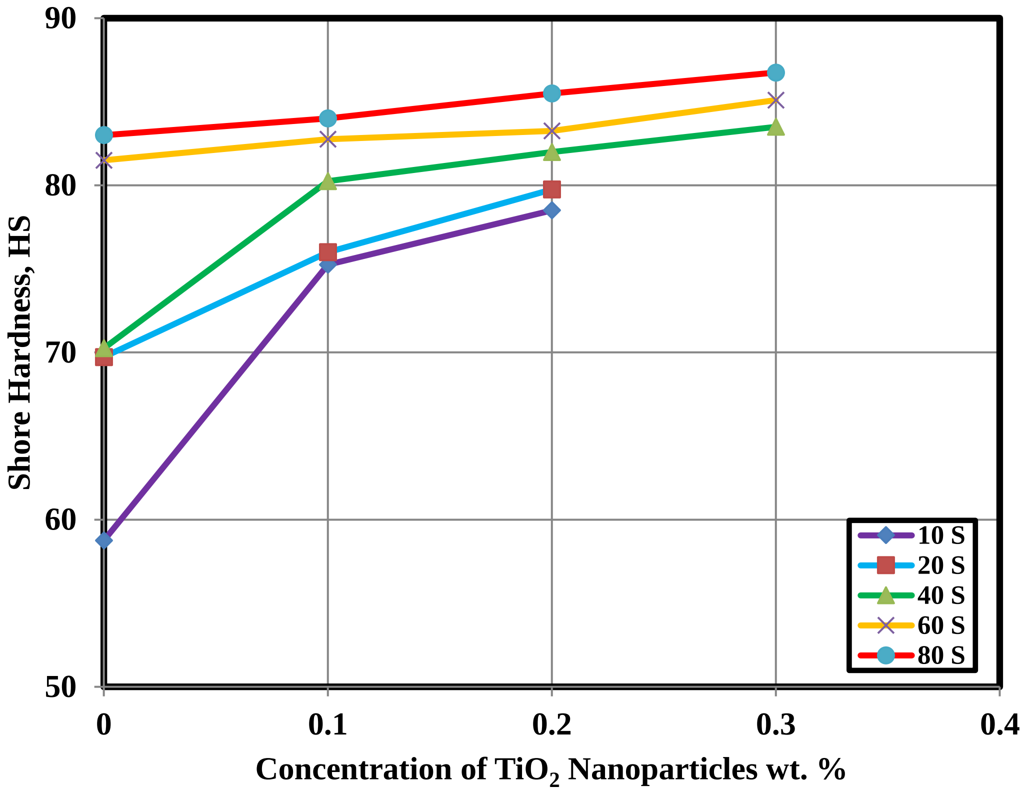


Fig. 11 Relationship between hardness and concentration of TiO_2 nanoparticles wt. % for gradually strong mode.

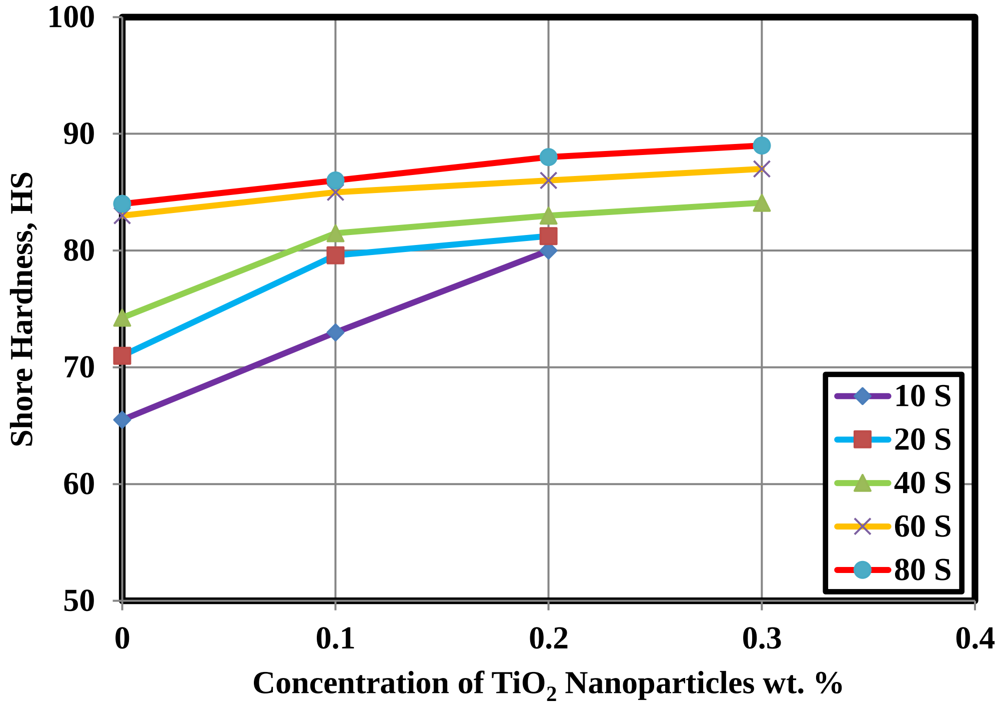


Fig. 12 Relationship between hardness and concentration of TiO_2 nanoparticles wt. % for flashing mode.

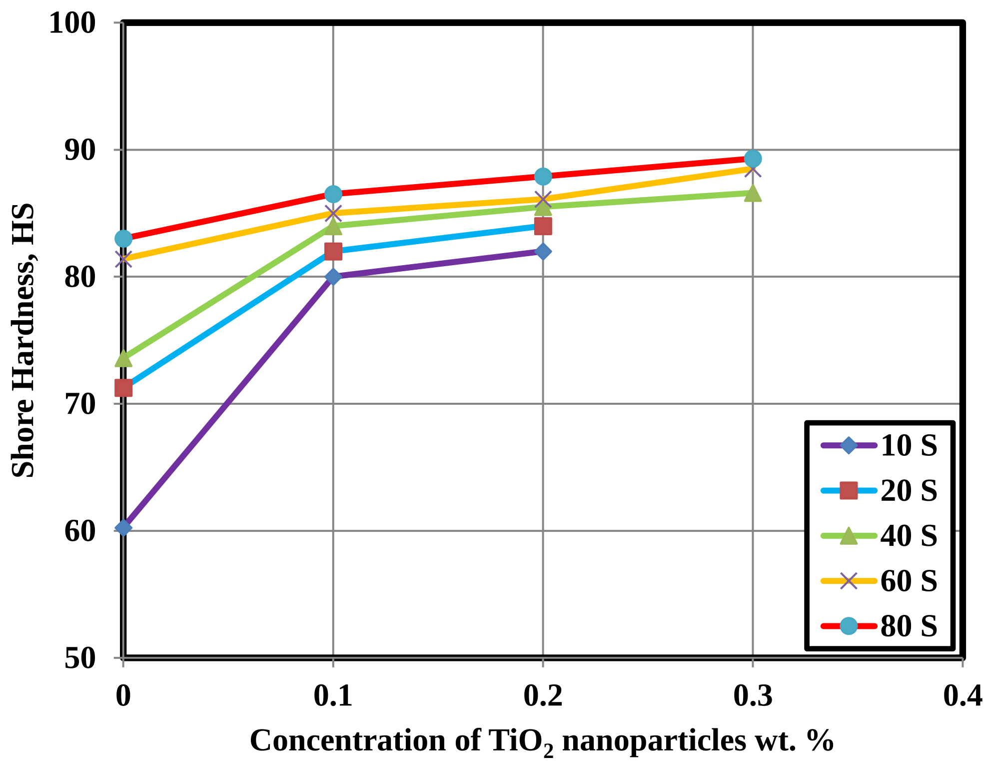


Fig. 13 Relationship between hardness and concentration of TiO_2 nanoparticles wt. % for strong mode.

Figure 12 shows the variation of Shore hardness of tested materials against concentrations of TiO_2 nanoparticles at 10, 20, 40, 60 and 80 seconds curing time. From Fig. 12, it is clear that Shore hardness of the tested materials increases with increasing curing time and the concentration of TiO_2 nanoparticles. Shore hardness at 80 seconds curing time increased by 18 % at 0.1 % TiO_2 nanoparticles compared to curing time 10 seconds. Values of Shore hardness of curing time 80 seconds are higher than other curing times.

Shore hardness of hybrid composite resin and nanocomposite resin against concentrations of TiO_2 nanoparticles at 10, 20, 40, 60 and 80 seconds curing time is represented in Fig. 13. It is clear that Shore hardness of the tested materials increases with increasing curing time and the concentration of TiO_2 nanoparticles. The obtained results as shown in Fig. 13 revealed that higher hardness values are attributed to high curing times. The tested materials cured for 80 seconds exhibited a higher Shore hardness compared to other curing times. Shore hardness at 80 seconds curing time is increased by 8 % at 0.1 wt. % TiO_2 nanoparticles compared to curing time 10 seconds.

Based on these results, it may be concluded that Shore hardness results of hybrid composite resin and nanocomposite resin are significantly affected by curing times. High curing times give higher hardness values compared to low curing times. Dentists can use 80 seconds curing time using the light emitting diode (LED) source for curing the tested materials used in this study.

CONCLUSIONS

Within the scope of work of the present study, the results showed that, Shore hardness of the hybrid composite resin and dental nanocomposite resin depended on the curing modes and curing times, the following conclusions can be drawn:

1. Gradually strong curing mode produced hybrid composite resin and dental nanocomposite resin with lower hardness.

2. Hardness results of specimens polymerized by strong curing mode are similar to these polymerized by flashing curing mode at 60 and 80 seconds for 0.1 and 0.2 wt. % TiO_2 nanoparticles groups.

3. Strong curing mode can be recommended as the best technique for curing the dental nanocomposite resin.

4. Curing times have significant influence on Shore hardness results of hybrid composite resin and nanocomposite resin.

5. High curing times give higher hardness values compared to low curing times.

6. Dentists can use 80 seconds curing time using LED source for curing the hybrid composite resin and nanocomposite resin.

REFERENCES

1. Annie J. St-Georges, Edward J. Swift Jr., Jeffrey Y. Thompson, Harald O. Heymann, "Irradiance Effects on the Mechanical Properties of Universal Hybrid and Flowable Hybrid Resin Composites", Dental Materials, Vol. 19, pp. 406–413, (2003).

2. Aljosa I., Tijana L., Larisa B., Marko V., "Influence of Light-Curing Mode on the Mechanical Properties of Dental Resin Nanocomposites", Procedia Engineering, Vol. 69, pp. 921 – 930, (2014).

3. Xia Y., Zhang F., Xie H., Gu N., "Nanoparticle-Reinforced Resin-Based Dental Composites, Journal of Dentistry", Vol. 36, pp. 450–455, (2008).

4. Marchan S., White D., Smith W., Coldero L., Dhuru V., "Comparison of the Mechanical Properties of Two Nano-Filled Composite Materials",Rev Clín Pesq Odontol, Vol.5 No. (3), pp. 241-246, (2009).

5. Ceballos L. , Fuentes M. , Tafalla H. , Martínez A., Flores J., Rodríguez J. , "Curing Effectiveness of Resin Composites at Different Exposure Times using LED and Halogen Units", J Clin Exp Dent., Vol. 1 No. (1), pp. 8-13,(2009).

6. Ronald L. Sakaguchi, John M. Powers, "Craig's RestorativeDental Materials", Elsevier, P. 190, (2012).

7. Aravamudhan K., Floyd CJ., Rakowski D., Flaim G., Dickens SH., Eichmiller FC, Fan PL., "Light-Emitting Diode Curing Light Irradiance and Polymerization of Resin-Based Composite", J Am Dent Assoc, Vol. 137, pp. 213-223, (2006).

8. Saade E., Bandeca M., A. N. S. Rastelli A., Bagnato V., and Porto-Neto S., "Influence of Pre-Heat Treatment and Different Light-Curing Units on Vickers Hardness of a Mcrohybrid Composite Resin, Laser Physics", Vol. 19, No. 6, pp. 1–6, (2009).

9. Hofmann N., Hugo B., Klaiber B., "Effect of Irradiation Type (LED or QTH) on Photo-activated Composite Shrinkage Strain Kinetics, Temperature rise, and Hardness", Eur J Oral Sci, Vol. 110, pp. 471-479, (2002).

10. Sakaguchi R., Berge H., "Reduced Light Energy Density Decreases Post-Gel Contraction while Maintaining Degree of Conversion in Composites", J Dent, Vol. 26, pp. 695-700, (1998).

11. García A., Lozano M., Vila J., Escribano A., Galve P., "Composite resins. A review of the Materials and Clinical Indications", Med Oral Patol Oral Cir Bucal, Vol. 11, pp. 215-20, (2006).

12. Leprince G., Palin W., Hadis M.,Devaux J., Leloup G., "Progress in Dimethacrylate Based Dental Composite Technology and Curing Efficiency", Dental Materials, Vol. 29, pp. 139-156, (2013).

13. Elsaka S. H., Hamouda I., Swain M., "Titanium Dioxide Nanoparticles Addition to a Conventional Glass-Ionomer Restorative: Influence on Physical and Antibacterial Properties", Journal of Dentistry, Vol. 39, pp. 589 – 598, (2011).

14. Sun J., Forster A., Johnson P., Eidelman N., Schumacher G., Zhang X., Wu W., Quinn G., "Improving Performance of Dental Resins by Adding Titanium Dioxide Nanoparticles", Dental Materials, Vol. 27, pp. 972–982,(2011).

15. Yu B., Ahn J., Lim J., Lee Y., "Influence of TiO_2 nanoparticles on the Optical Properties of Resin Composites", Dental Materials", Vol. 25, pp. 1142–1147, (2009).

16. ASM International, ASM Handbook Volume 8, "Mechanical Testing and Evaluation", American Society for Metals, Metals Park, Ohio, P. 554, (2000).

17. Pontes L., Alves E., Ballester R., Alves B., Dias C., Silva C., "Mechanical Properties of Nanofilled and Microhybrid Composites Cured by Different Light Polymerization Modes", General Dentistry, pp. 30-33, (2013).