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Influence of Light-Curing Mode on the Mechanical Properties of Dental Resin Nanocomposites

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Abstract

Dental clinicians need to consider the engineering properties of the dental restorative materials during creating the restoration plan that provide the best therapeutic outcome. In the oral environment, dental materials are exposed to various physico-chemical, biological and mechanical challenges. Dental materials have to be sufficiently mechanically resistant to withstand the high occlusal forces that occur in the mouth during chewing. These forces can reach the values of 200 N in the anterior occlusal region, and up to 800 N in the lateral occlusal segments, or even up to 3500 N during some abnormal jaw movements and teeth contacts. Dental resin-based composites are most widely used materials for dental restoration. There has been a lot of research effort to improve organic and inorganic dental composite formulations in order to produce resistant aesthetic material. Different light-curing processing conditions have been proposed, in order to improve degree of monomer conversion, to reduce polymerization shrinkage and stresses, and to improve the mechanical characteristics of the material. The aim of this study was to determine the influence of two different light-curing protocols on the mechanical properties of three types of contemporary dental resin-based composites. Experiments were conducted in the laboratories of the University of Novi Sad.

Keywords: Mechanical properties; polymerization modes; dental resin-based composites; compressive strength; diametral tensile strength; Vickers hardness

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1. Introduction

Therapeutic success in clinical restorative dentistry depends not only on understanding of biological, physiological and pathophysiological principles, but on a complete understanding of restorative materials properties and the therapeutic procedures [1]. Only integrative knowledge of the dental operative principles and the materials engineering properties can lead to the proper material choice in particular clinical situation [2].

The most commonly used materials in contemporary restorative dental practice are resin-based composites [3]. Dental composites are tooth-coloured materials that consist of organic resin matrix (dimethacrylate or silorane resins), inorganic fillers (zirconium-dioxide, silicium-dioxide and other glass particles), organo-silane coupling agent, and photo-initiators and accelerators [4]. These materials are based on photo-cured resin monomers, which harden by exposing to visible light energy source.

The mechanical properties of dental restorative materials are very important for their clinical performance and duration. Influence of photoactivation method on mechanical properties of resin based materials was presented in paper [5]. Dental restoratives need to withstand the high occlusal forces that occur in the oral environment, and to be resistant to ensure stable therapeutic success. Biting forces in oral system reach values of about 200 N in the frontal masticatory region, up to 800 N in the lateral occlusal segments, or even up to 3500 N during some abnormal jaw movements and teeth contacts [1,6]. The mechanical properties of dental composites depend on the resistance of its organic and inorganic components and on the bond between these phases [7]. Polymer structure and the degree of conversion are important properties that can influence the overall materials characteristics [8]. These properties depend not only on the resin structure, but also on the type and distance of the light-curing source, on the light irradiance, the exposure time, the mode of photo-polymerization procedure etc. [9]. The spatial structure of polymer molecules can be affected by altering the light-curing polymerization procedure (Fig. 1) [1]. In the past years, many different light-curing modes have been introduced in dental practice, mainly in order to reduce the degree of polymerization shrinkage of the composite materials. There was a need not to disturb the mechanical strength during such light-curing protocols and to find working conditions that allow the best therapeutic results [9].

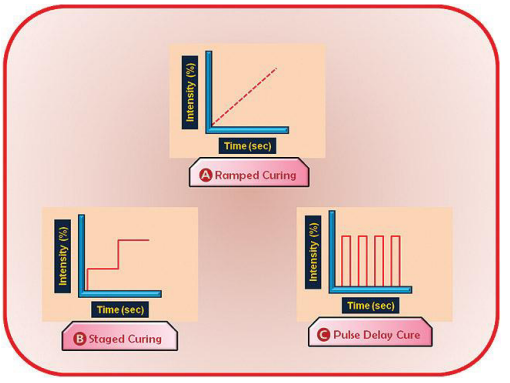

Fig. 1. Different dental light-curing modes [10].

On the other hand, the inorganic filler type and content have the major influence on the composites mechanical behavior [11]. Dimensions of filler particles have been changed and reduced over the years, down to the nanoparticles, in order to improve materials characteristics [4,12]. Nanoparticles have high surface area and unique physicochemical properties, and they can influence the overall bulk material features [12,13,14]. Further, high inorganic filler fraction decreases the volume of the organic matrix that cause negative material properties, such as: polymerization contraction, high coefficient of thermal expansion, sorption and solubility, low mechanical strength, low wear resistance etc [15,16,17].

In every-day dental clinical practice, there is a need for using the best possible dental processing methods in order to utilize the best materials features. It was important to investigate, what is the effect of dental ramped

photoactivation mode on mechanical strength of contemporary dental nanomaterials, comparing to the effect of conventional photoactivation mode on these properties.

In this study, we have tested the influence of two different light-curing protocols on the mechanical properties of three types of contemporary dental resin-based composites.

Nomenclature

p	compressive strength [N/mm^2]
F_{CT}	force occurred in compressive test processes [N]
D_{CT}	diameter of specimens used in compressive test [mm]
DTS	Diametral tensile strength [N/mm^2]
F_{DTS}	force occurred in diametral test processes [N]
D_{DTS}	diameter of specimens used in diametral tensile test [mm]
T	thickness of the specimens used in diametral tensile test [mm]
HV	Vickers hardness [HV]
F_V	force applied in Vickers test process [N]
d_1,d_2	imprint diagonals of Vickers indenter [mm]

2. Materials and methods

Three contemporary dental resin-based composites were tested in this study. Each tested material represents specific type of composite with different inorganic filler compositions (nanofilled, nanohybrid and microhybrid material). Detailed informations about the materials used in the study are shown in the tables 1, 2 and 3.

Specimens of each material were made by using cylindrical molds made by Rapid prototyping technology. Dimensions of specimens were ϕ4x4mm for compressive test, ϕ5x2mm for diametral tensile test and ϕ4x2mm for Vickers hardness test. Molds were placed on the glass microscope slide, filled with material and covered with another glass slide, taking care to obtain a flat surface without any defects and entrapped air. Material was then polymerized by exposing to the light of a dental light-energy source (Bluephase® C8, Ivoclar Vivadent – Fig. 2).

Table 1. Details about Filtek Ultimate Body tested in study.

Name	Filtek Ultimate Body (FBU)
Manufacturer	3M ESPE, St. Paul, MN, USA
Classification	Nanofiled
Lot no	N349776
Shade	A2
Matrix	Bis-GMA, UDMA, Bis-EMA, TEGMA and PEGDMA
Fillers	non-agglomerated/non-aggregated 20 nm silica filler, non- agglomerated/non-aggregated 4-11 nm zirconia filler, and aggregated zirconia/silica cluster filler (average cluster particle size – 0,6-10 μm)
Filler loading	78,5wt%, 63.3 vol%

Two different polymerization protocols were applied: conventional and soft start mode.

Conventional light-curing protocol means emitting of a constant, stable, full-intensity light irradiance of 800 mW/cm^2 throughout the whole polymerization cycle. Soft start polymerization protocol means emitting of a reduced light energy at the beginning of polymerization cycle (650 mW/cm^2), followed by a full light-energy intensity (800 mW/cm^2). Detailed informations about polymerization modes are shown in table 4.

For each mechanical test in this study, six specimens of each material were used, three polymerized using conventional curing protocol and three polymerized using soft start curing protocol.

Experimental tests were conducted in the Laboratory for Materials on Faculty of Technical Sciences. Mechanical press with 50KN rated force was used for compressive test and diametral tensile test. Force transducer, displacement transducer and Spider8 universal amplifier were used for precise force and stroke measurement. Vickers hardness testing machine was applied for the hardness test.

Table 2. Details about Filtek Z550 tested in study.

Name	Filtek Z550 (FZ550)
Manufacturer	3M ESPE, St. Paul, MN, USA
Classification	Nanohybrid
Lot no	N340139
Shade	A2
Matrix	Bis-GMA, UDMA, Bis-EMA, TEGMA and PEGDMA
Fillers	Surface-modified zirconia/silica fillers 3000 nm (3 μm or less), non-agglomerated/non-aggregated surface-modified silica particles 20 nm
Filler loading	82 wt%, 68 vol%

Table 3. Details about Filtek Z250 tested in study.

Name	Filtek Z250 (FZ250)
Manufacturer	3M ESPE, St. Paul, MN, USA
Classification	Microhybrid
Lot no	N367949
Shade	A2
Matrix	Bis-GMA, UDMA, Bis-EMA, TEGMA
Fillers	Zirconia, silica 10 – 3500 nm (0,01-3,5 μm)
Filler loading	75-85 wt%, 60 vol%

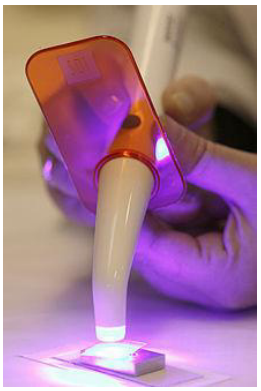

Fig. 2. Polymerization of the specimens by exposing to light of dental light-energy source [18].

Table 4. Details about tested photoactivation modes.

Photoactivation mode	Irradiance (mW/cm^2)	Polymerization duration
Conventional	800 mw/cm^2	40 seconds
Soft Start	650 mW/cm^2 followed by 800 mW/cm^2	40 seconsd

2.1. Compressive test

Compressive test was conducted according to figure 3. Dimensions of specimens were ϕ4x4mm and upsetting was carried out using flat dies. Press velocity was 10mm/min.

Process was conducted until the crack. No lubrication was applied. Figure 4 presents specimen before and after experiment. Compressive strength p was calculated according to equation (1).

$$p = \frac{4F_{CT}}{D_{CT}^2 \pi}\left[\frac{N}{mm^2}\right] \tag{1}$$

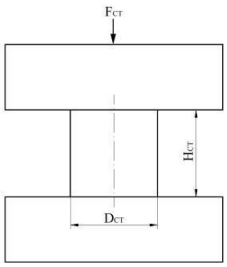

Fig. 3. Compressive test procedure.

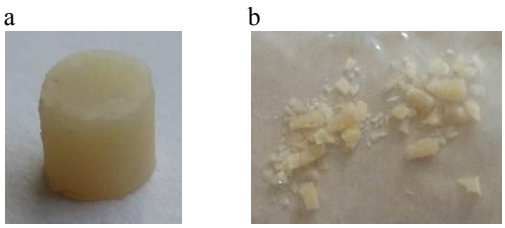

Fig. 4. Specimens before (a) and after (b) experiment.

2.2. Diametral tensile test

Diametral tensile test is a common method for measuring tensile strength of brittle materials because it avoids some of the difficulties inherent in direct and flexural tensile testing [19].

In the diametral tensile test (*DTS) s*pecimens were compressed diametrically introducing tensile stress in the material in the plane of the force application, figure 5.

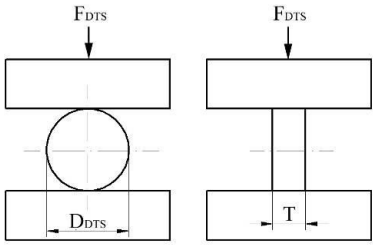

Fig. 5. Diametaral tensile test procedure.

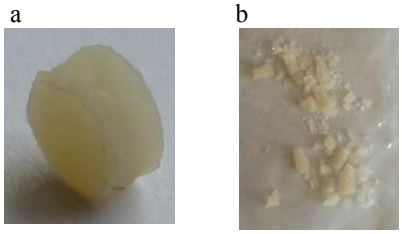

Fig. 6. Specimens before (a) and after (b) experiment.

Dimensions of specimen were ϕ5x2mm. In figure 6 one specimen before and after the experiment is presented. No lubrication was applied. Press velocity was 10mm/min. Compression was carried out using flat dies until the crack. Calculation of *DTS* was conducted according to equation (2).

$$DTS = \frac{2F_{DTS}}{D_{DTS}\pi T}\left[\frac{N}{mm^2}\right] \tag{2}$$

2.3. Vickers hardness test

For Vickers hardness test specimens were ϕ4x2mm, figure 7.

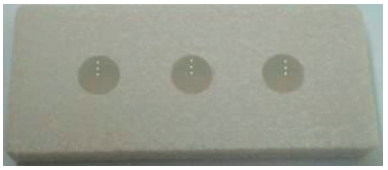

Fig. 7. Vickers hardness test specimens.

In this test, due to easier positioning on the test machine, specimens were remained in the moulds. Hardness was measured on both sides of the specimens (TOP and BOTTOM) using diamond indenter in the form of a right pyramid with a square base and an angle of 136 degrees between opposite faces (figure 8), subjected to a load of 98.1N. Loading time was 30s. Six measurements per specimen were carried out, three on top and three on bottom side of each specimen. In figure 9 imprint of diamond indenter is presented.

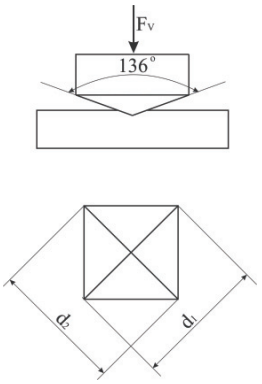

Fig. 8. Hardness test procedure.

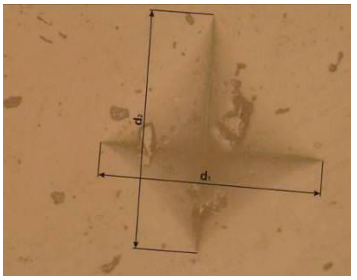

Fig. 9. Diagonals of imprint.

Diagonals d_1 and d_2 were implemented in equation (3) which was used for hardness calculation.

$$HV = 0{,}1891\frac{F_V}{\left(\frac{d_1 + d_2}{2}\right)^2}\left[\frac{N}{mm^2}\right] \tag{3}$$

3. Results

The objective of this experimental program was to investigate influence of polymerisaton mode on mechanical properties of specimens. Standard and soft start polymerization modes were used. Compressive test, diametral tensile test and hardness test were conducted on three different dental materials.

Forming-stroke diagrams for compressive test and diametral tensile test are presented in figures 10 – 15. For Filtek Ultimate Body and Filtek Z250 higher values of force have occurred for standard polymerization mode. Soft Start mode gives higher force values for Filtek Z550 material.

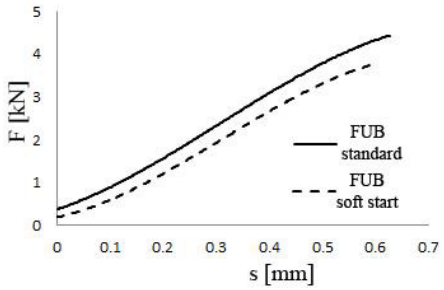

Fig. 10. F-s diagram in compressive test for FUB.

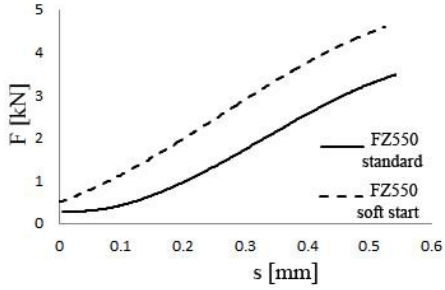

Fig. 11. F-s diagram in compressive test for FZ550.

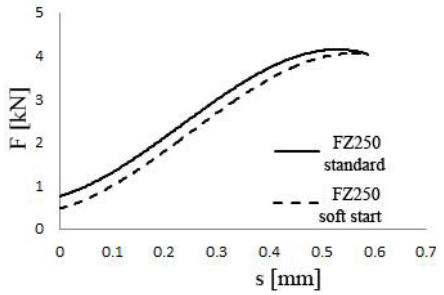

Fig. 12. F-s diagram in compressive test for FZ250.

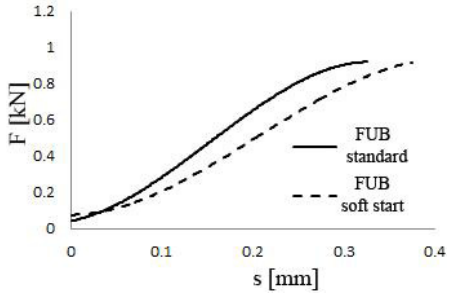

Fig. 13. F-s diagram in diametral tensile test for FUB.

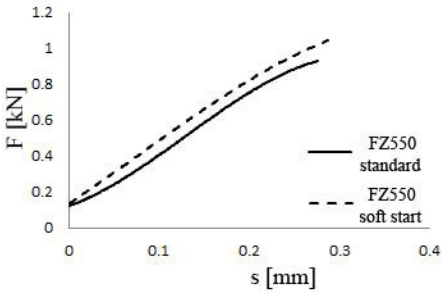

Fig. 14. F-s diagram in diametral tensile test for FZ550.

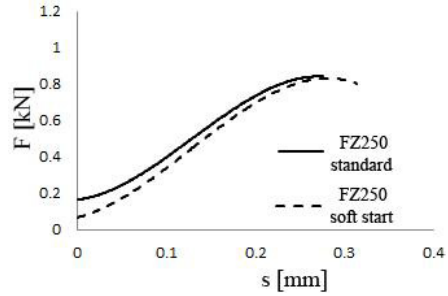

Fig. 15. F-s diagram in diametral tensile test for FZ250.

In figures 16 - 18 values of compressive strength for different materials are presented. It can be seen that in case of Filtek Ultimate Body (FUB, figure 16) and Filtek Z550 (FZ550, figure 17) values of compressive strength p was

higher for soft start polymerisation mode. However, value of *p* was lower for soft start method in case of material Filtek Z250, figure 18.

Values of diametral tensile strength *DTS* are presented in figure 19 - 21. As it can be seen in case of Filtek Ultimate Body (figure 19) diametral tensile strength was higher for soft start polymerization mode. Soft start mode gives lower values of *DTS* in case of FZ550 and FZ250, figure 20 and 21.

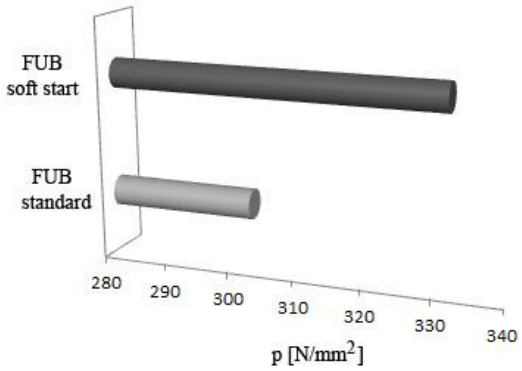

Fig. 16. Compressive strength for FUB.

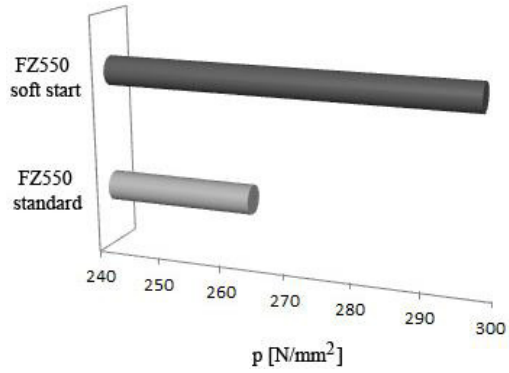

Fig. 17. Compressive strength for FZ550.

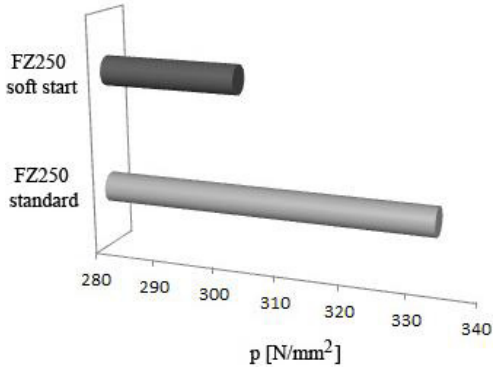

Fig. 18. Compressive strength for FZ250.

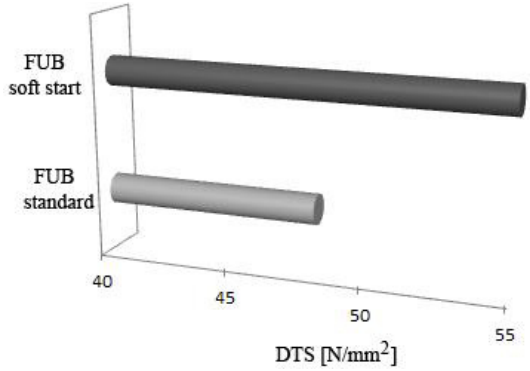

Fig. 19. Diametral tensile strength for FUB.

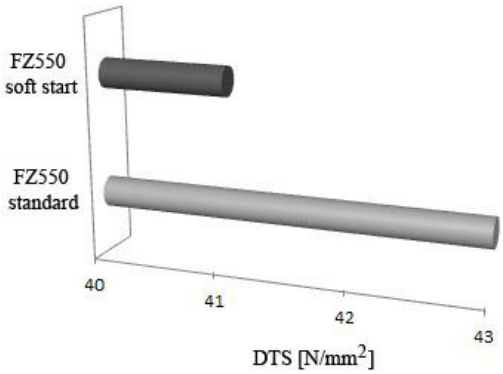

Fig. 20. Diametral tensile strength for FZ550.

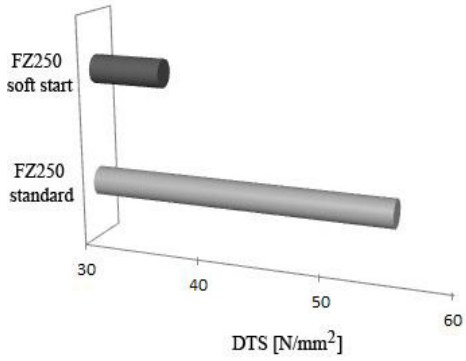

Fig. 21 Diametral tensile strength for FZ250.

Results obtained by Vickers hardness test are shown in figures 22 - 24. For each material and polymerization mode hardness was measured on top and bottom side of the specimens. From figure 22, it can be seen that conventional curing protocol produced FUB material with lower hardness values, on the both – top and bottom sides of the specimens. In this study, Filtek Ultimate Body showed complete sensitivity to light-curing protocol through the all three used mechanical tests. Soft start curing mode can be proposed as a recommended method for polymerizing this material.

The Filtek Z550 specimens (figure 23) showed higher values of hardness on the top side when they have been polymerized by soft start curing mode, but on the bottom side, hardness values were higher for specimens cured by conventional protocol.

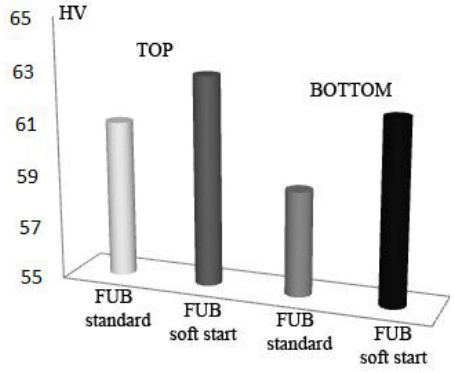

Fig. 22. Hardness test values for FUB.

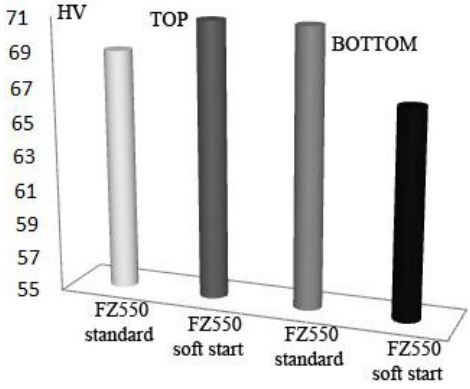

Fig. 23. Hardness test values for FZ550.

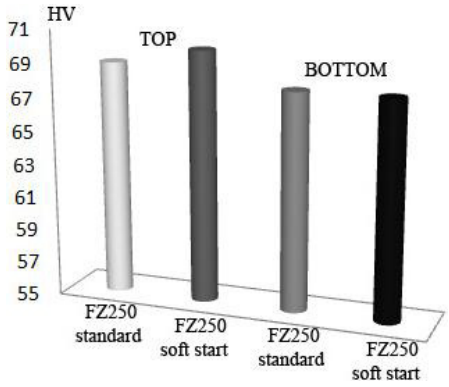

Fig. 24. Hardness test values for FZ250.

Soft start curing protocol produced higher hardness values on the top sides of the Filtek Z250 specimens (figure 24). The similar hardness results were obtained between specimens polymerized by conventional and soft start curing protocol on the bottom surfaces of the Filtek Z250 specimens.

The hardness values on the top of the specimens present direct correlation with degree of monomer conversion [9].

During polymerization cycle, light was partially scattered and absorbed through the depth of the specimens, which can explaine slightly lower bottom hardness values. It can be seen, that some inversions of the top and the bottom harness values appeared for the both curing protocols. These results show that polymerization is not homogeneous through the whole specimen, and that it is not simply spatially distributed.

4. Conclusion

Within the limitations of this study, it can be concluded that the influence of the light-curing protocol on mechanical properties is material- and property- dependent. The soft start photo-polymerization protocol mostly produced higher hardness values of all tested dental materials. These results indicate that this curing mode produce composites with sufficient degree of monomer conversion, and perhaps even the material with more quality polymer network formation. Further research need to define and make it clear, what polymerization protocol is suitable for which dental composite material and to create precise recommendations for clinical work. Further, future investigations should focus on the overall assessment of the effects of certain polymerization methods on materials properties, and to define the optimal materials properties wanted to be achieved considering the oral environment conditions.

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