The difference nanocomposite hardness level using LED photoactivation based on curing period variations

Hasiana Tatian*), Moch. Richata Fadil*, Milly Armilia*

***Department of Conservative Faculty of Dentistry Universitas Padjadjaran**

ABSTRACT

Polimerizatian is the critical stage to determined the quality of composites resin, this involves isolated monomer carbon double bonds being converted to an extended network of single bonds. Physical and mechanical properties of composites are influenced by the level of conversion attained during polymerization. An adequate light intensity and light curing time are important to obtain degree of polymerization. The objective of this study is to evaluate the difference of the hardness nanocomposites which activated by LED LCU based on variation of curing times. This study is a true experimental research. The samples was made from nanocomposites material with cylinder form of 4 mm in depth, 6 mm in diameter. This samples divided into 3 groups of curing times. Group I was cured for 20's curing time as control due to manufactory recommended; Group II was cured for 30's; and Group III was cured for 40's and the hardness (Rebound hardness tester) was determined using Rebound scale (RS) and converted by Mohs scale (MS). There were very significant level of hardness rate from each group using Anova test. The result of the study conclude that there was the differences on the nanocomposites hardness level cured under different curing times 20, 30 and 40 sec. The longer of curing times, the higher level of hardness.

Key words: Degree of conversion,nanocomposite, photoactivation, LED LCU, curing

ABSTRAK

Proses polimerisasi merupakan tahap yang kritis bagi penentuan kualitas material resin komposit, karena pada peristiwa polimerisasi ikatan karbon ganda monomer berkonversi menjadi ikatan karbon tunggal membentuk rantai polimer. Derajat konversi yang tinggi menghasilkan komposit yang memiliki sifat fisik mekanik yang tinggi. Intensitas cahaya yang memadai serta lamanya waktu curing berpengaruh besar dalam peningkatan derajat polimerisasi. Penelitian ini bertujuan untuk melihat perbedaan tingkat kekerasan nanokomposit yang dilakukan fotoaktivasi dengan LED LCU berdasarkan variasi waktu curing. Metode yang digunakan dalam penelitian ini adalah eksperimental murni. Sampel dibuat dari bahan Nanokomposit berbentuk silinder dengan diameter 6 mm dan kedalaman 4 mm. Sampel ini dibagi dalam 3 kelompok perlakukan waktu curing. Kelompok I dengan waktu curing 20 detik, sesuai aturan pabrik sebagai kontrol; kelompok II, 30 detik; dan kelompok II, 40 detik. Penelitian dilakukan dengan Rebound hardness tester. *Hasil penelitian diuji secara statistik menggunakan uji Anova. Hasilnya terdapat perbedaan tingkat kekerasan yang sangat signifikan dari setiap kelompok. Simpulan dari penelitian ini*

*)*Correspondence author: Hasiana Tatian, Department of Conservative Dentistry Faculty of Dentistry Universitas Padjadjaran Jl. Sekeloa Selatan No. 1 Bandung, West Java-Indonesia, Tel./Fax: +6222-2504985/2532805*

adalah terdapat perbedaan tingkat kekerasan nanokomposit yang dilakukan curing dengan waktu 20, 30, dan 40 detik. Semakin lama waktu curing, semakin tinggi tingkat kekerasannya.

***Kata kunci**: Tingkat kekerasan, nanokomposit, fotoaktivasi, LED LCU, curing*

INTRODUCTION

Composite is an aesthetic restoration material used in wide scaled conservation field. Its physical and mechanical abilities yield various chances and the choice of material can be used in various circumstances. Knowledge mastery of the natures of composite allows the operator to work and manipulate in accordance with the limitations so that the shortcomings of this material can be overcome and gain a good restoration result.[1,2]

Since first discovered, research and development on this material has been continued in order to gain better physical, mechanical, and aesthetical natures, and to make it easily handled.[3,4] Formula repair, the newest technique development such as nano technology application makes direct composite restoration more reliable.[5-7]

Adequate curing will produce a composite that is durable to chemical changes, owns strong and hard mechanic properties. The hardness is influenced by conversion degree which is determined by the quality of photo activation source.[8,9] In the process of polymerization, light intensity and curing time are two important factors that are highly influential in the delivery of a number of photons to specimen.[10]

Light emitting diode light curing units (LED LCU) is a photopolymerization source that has long edge wave matches camphorquinone photo initiator absorption spectrum as the common photo initiator used[11,12], one of LED LCU positive side is, it can be operated without a significant intensity declination and it owns constant light output power and spectrum.[11,13]

METHODS

The type of research used was pure experimental research. The sample was nanocomposite restoration material with the following criteria: (1) The composition of resin system: BIS-GMA, BIS-EMA (6), UDMA, TEGDMA; (2) Filler: a mixture of non group nano silica filler with the filler size 20 nm; grouped of loose bond of zirconium particle and nano cluster silica size 0.6-1.4 μ with the original particle size 5-20 nm; (3) Material from one product with the same type and manufacture date; and the color was A3.

The research samples was a composite molded in a cylindrical mold with a diameter of 6 mm and a depth of 4 mm. Samples were made in 30 pieces, divided into 3 groups, each 10 pieces.

The materials used in this research were resin composite type nano composite and cellulose strip. The tools used in this research were Ash 29, large and small cement stopper, sonde, glass lab, mold for molding specimen samples, marker pen for marking the top part of the samples, Light curing unit type high power LED, and Rebound hardness tester.

Samples were made with a mold made of metal plate. It was aimed to make the light focused on the composite which will be polymerized and not to make it contaminated by other lights from the outside that will affect the polymerization process. The mold was in the form of a cylindrical hole with the diameter of 6 mm so that it would be in the circle of LED LCU polymerization area with the diameter of 8 mm. The depth of the mold was 4 mm that assumes the average depth of dental caries. The center of the metal was cut in order to facilitate sample excretion. The top and bottom parts of the metal were covered by a fiber plate in order to produce a mold with flat and smooth surface which is needed for sample placement in the hardness measurement table.

Mold was equipped with a suppressor with a constant and certain weight so that each sample gets the same pressure, and there were 2 disassembled pins. The pins were plugged in and penetrate the top fiber plate, metal mold, bottom fiber plate and the suppressor table. The function of the pins was to make the mold position always in the same place and position in every mold making as well as for mold stability during suppression time.

Curing process, each group consists of 10

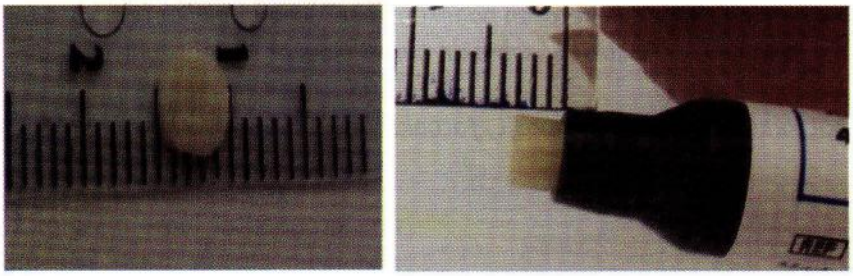

Figure 1. A. composite diameter measurement; B. composite length measurement to put in the mold.

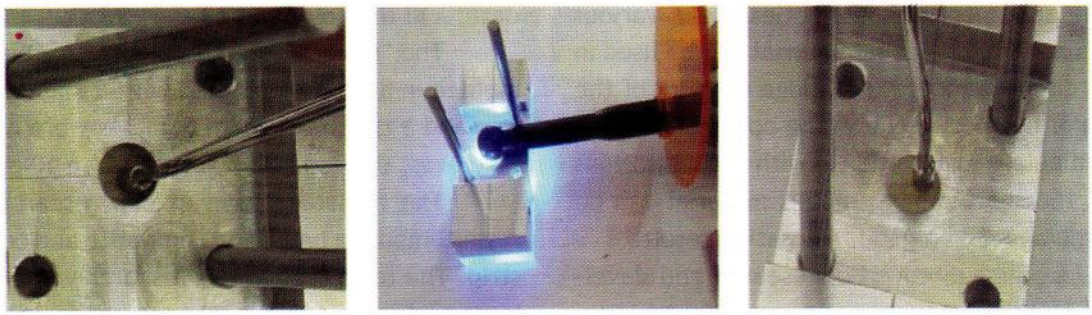

Figure 2. A. Composite is put into the mold with 2 mm layer, compacted; B. Composite is polymerized; C. The second layer.

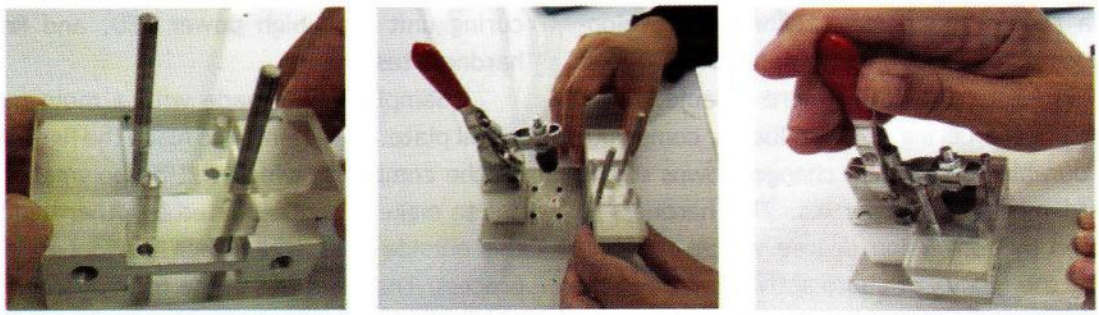

Figure 3 A. Sample is covered by a cellulose strip, the upper fiber plate is installed; B. Mold is placed under the suppressor; C. Mold is pressed for 5 seconds.

samples. Group I with 20 second curing time is recommended by the factory as the control, group II with 30 second curing time, and group IV with 40 second curing time. The making of the sample is started from the 20 second time group and so on.

The samples were coated twice, each with 2 mm thickness. The excreted composite from the tube was measured in length and diameter so that the first layer in the mold gets 2 mm thickness (Fig. 1 A and B), the calculation formula was provided in the attachment.

Composite was inserted into the compressed mold with a stopper until it was really solid and then was irradiated, followed immediately by the second layer, compressed with a stopper until it fits the mold, then the upper part was covered by a cellulose strip.

Fiber plate was placed on the mold and was put under a suppressor. As it is pressed with the suppressor, leave it for 5 seconds, and then the suppressor was opened, the upper part of the fiber was also opened; composite excess comes out as excess. The cellulose strip was in its position to cover the composite in order to prevent the production of oxygen barrier layer (Fig. 1A).

The composite was polymerized on the cellulose strip (Fig. 1B), the cellulose strip was then removed, and then the upper part of the sample was marked (Fig. 1C) in order to show the upper part of the mold which was close to the light, and also the serial number was written on the front of the sample. The sample was removed from the mold, cleaned from the excess, and lastly it was ready to be tested (Fig. 2 A and B).

Samples are made in 30 pieces and divided into 3 irradiation time group, each group consists

Figure 4 A. Sample was covered by a cellulose strip, the upper fiber plate was installed; B. Mold was placed under the suppressor; C. Mold was pressed for 5 seconds.

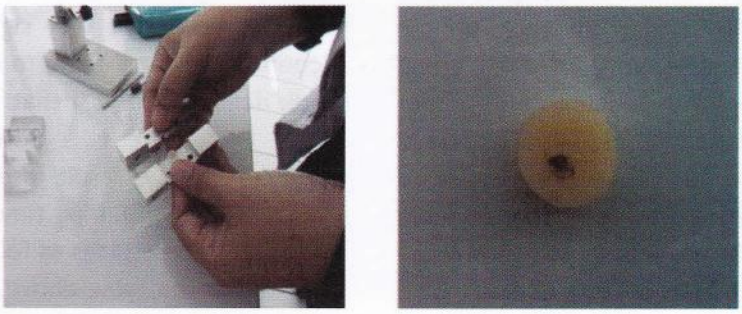

Figure 5. A. Suppression excess; B. Composite was polymerized (cellulose strip was not opened); C. The marking of the upper part of the sample (which was near to the light).

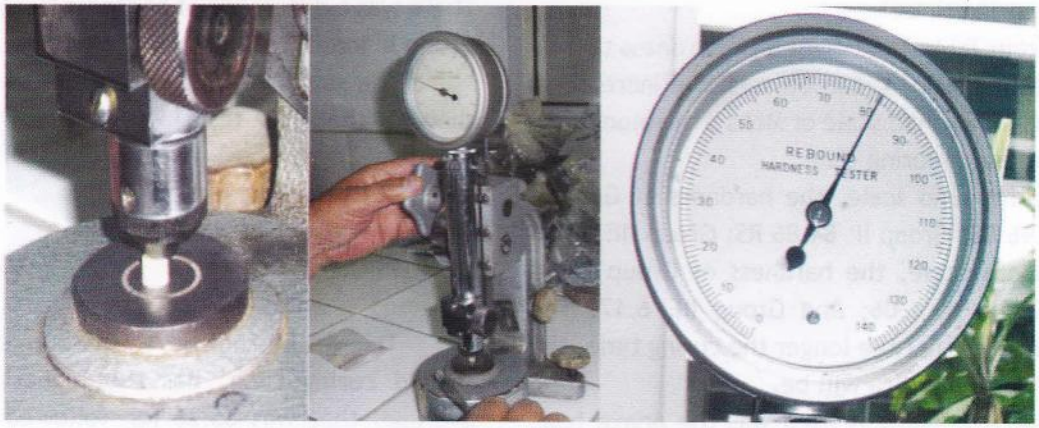

Figure 6. A. Mold was removed; B. Sample was ready to be tested.

of 10 samples. Group I with 20 second curing time as recommended by the manufacturer as the control; Group II with 30 second curing time; and Group III with 40 second curing time.

The samples going to be tested were stored in 3 plastic bags according to each group, and then they are stored in containers protected from light, and were opened when the hardness test was performed. The period of the first sample making until the last one was 24 hours.

Hardness test on each sample was carried out on the marked area. Each area had 5 points. The test was according to the group and serial number.

The sample was placed on the rebound hardness tester instrument test table, set the clamp so that the sample was in stable position, not moving (Fig. 3A), hitter button was activated (Fig. 3B), reflection scale result (Fig. 3C), seen in dial and recorded as the test point 1 serial number 1 Group I.

Next, the second test point was carried out by shifting the sample a little bit from the first test point position by doing the same test as test point 1, test point 2 serial number 1 Group 1 was obtained, continued until test point 5. The test ended at test point 5 serial number 10 Group III.

Table 1. Hardness test result curing time 20 seconds

Sample	1	2	3	4	5	6	7	8	9	10
Group I	81.20	79.80	81.40	80.60	80.20	80.20	80.80	81.80	82.80	81.60
Group II	85.20	82.60	85.00	84.20	85.80	85.60	85.20	84.80	84.80	84.60
Group III	86.20	85.80	86.40	86.40	86.40	86.00	87.20	86.20	86.00	87.00

RESULTS

Research on nanocomposite hardness difference using LED photoactivation with curing time variations was performed on samples made of special molds. The data from each sample was recorded, hardness average of each group was calculated using the Rebound (RS) scale, converted into the Mohs (MS) scale and analyzed statistically.

The hardness test data can be seen in Table 1. The result shows the hardness' difference in rebound scale and Mohs scale and also there was a significant hardness' difference based on the nanocomposite statistic analysis result which is activated by LED between the 20, 30, and 40 second curing time.

From Table 1, the result of hardness test by curing time group, it can be seen the increase in hardness in rebound scale or Mohs scale along with the increase in curing time.

In rebound scale, the hardness of Group I was 80.96 RS; Group II: 84.86 RS; Group III: 86.38 RS. In Mohs scale, the hardness of Group I was 5.78; Group II, 6.06; and Group III, 6.17. The result showed that the longer the curing time, the harder the composite will be.

Statistic analysis used was the Anava test. Anava test aimed to find out if there was any difference in population average, whether or not there was any hardness' difference in the specimens treated with curing time of 20, 30, and 40 seconds.

Table 2. Multiple comparisons dependent variable: hardness test result

(J)Curing time	(J)Curing time	Mean Difference (I-J)	Sig.
20 Seconds	30 Seconds	-3.9400	0.000**
	40 Seconds	-5.4400	0.000**
30 Seconds	20 Seconds	3.9400	0.000**
	40 Seconds	-1.5000	0.000**
40 Seconds	20 Seconds	5.4400	0.000**
	30 Seconds	1.5000	0.000**

The test result obtained with the statistical package program SPSS Ver. 15 can be seen in the Table 2. From the Table 2, it can be seen that the test result was very significant ($p<0,000$) or F count 158,548> F table (α = 5%, dk numerator = 2, dk denominator 27) that was 4,91. This result shows that there is a difference between the hardness average result of each group caused by the treatment of curing time. The comparison of each hardness result is as follows:

From the table above, it can be concluded that: (1) There is a difference of hardness result between Group I and II ; (2) There was a difference of hardness result between Group I and III; (3) There was a difference of hardness result between Group II and III.

The statistic analysis result of the research showed that all tests were very significant, meaning there were a difference of hardness result between each group.

DISCUSSION

The result showed the Group II, with 40 second curing time, has the highest hardness average, that was 86.36 RS, followed by Group II, with 30 second curing time yields the hardness average of 84.86 RS and the lowest hardness average was produced by group I with 20 second curing time as recommended by the manufacturer, that was 80.92 RS.

If the hardness score was converted into the Mohs scale, the hardness of group with 40 second curing time was comparable with 6.17 MS. This score indicates 6-7 MS hardness which represents the hardness of glass mineral and quartz fused groups. The hardness of group with 30 second curing time was comparable with 6.06 MS, represents the hardness of orthoclase, titanium, manganese groups, etc. Group with 20 second curing time was comparable with 5.78 MS represents the hardness

between the apatite, zirconium, and orthoclase minerals.[14,15]

The result shows the longer the time for composite photoactivation, the highest the composite's hardness will be. This situation was possible because there are important factors in the process of composite polymerization, that was light intensity and curing time which has strong influence in the delivery of photons to specimens.[10]

The use of optimum curing method was very important, as several studies show the polymerization extension which directly related to the physical and biological natures. According to Rueggeberg, minimum light intensity which can be used for the polymerization of adequate resin composite with 2 mm deep layer is 280 milli/cm^2.[15] The LED LCU used in this research was included to the high power output class with light intensity over 730 mW/cm^2.

Curing time was one of important factors of several variables which regulate the polymerization of resin composite restoration material. If light intensity is enough to activate the camphoroquinone contained in resin composite, then curing time is the next step for polymerization process.[16]

LED LCU was the source of polymerization light which its intensity does not change and also had a narrow output spectrum around 440-490 nm that matches the camphorquinone absorption spectrum.[17] LED LCU light activation can make the camphorquinone remains activated or keeps being in the triplet phase and then was reacted with the amine reducting agent to form free radicals and begin the process of composite polymerization.[17,18]

Therefore, photo polymerization depends on irradiation intensity and curing time.[19] High light intensity and long curing time will result a high hardness score.[10] An adequate polymerization process was a fundamental phase for obtaining optimum physical, mechanical and chemical natures as required in an ideal clinical situation,[18] because during polymerization, it will occur double bond conversion of monomer aliphatic carbon into polymer single carbon bond which is known as conversion degree.[20]

Conversion degree is influenced by several factors including the curing time. According to Leung et.al. and Tarumi et.al, the polymerization reaction on composite activated by light will continue even until the end of irradiation, thus curing time must be used optimally by the clinicians in order to obtain high quality composite restoration.[21]

Twenty second curing time as recommended by the manufacturer may be a suggestion for the use of minimum curing time. Based on the research result, composite hardness increases with significant result if the curing time was extended 30 seconds and the most optimum was the 40 second curing time. It can be assumed that camphorquinone as a photosynthesizer in resin composite has not been used optimally if the curing time used was only 20 seconds, as well as the conversion degree resulted still can be increased.

A very significant hardness increase with 40 second curing time proves that there is an increase in the conversion degree of resin composite. The increase of conversion degree will decrease water sorption and composite's solubility which are related to the quality enhancement and longevity of compost resin.

CONCLUSION

Based on the collected data from the research result and the average calculation of the hardness level using the Rebound scale and statistic analysis, it can be concluded as follows: The hardness level of nanocomposite which is polymerized by light emitting diode light curing units is highly significantly different based on the curing time, the 20 second, 30 second irradiation, and 40 second irradiation. The longer the irradiation time is, the highest the hardness level will be.

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