Effects of light curing and remineralization on micro hardness of nano esthetic restorative materials

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Abstract: Objectives: The aim of this study was to investigate the effect of remineralization and light exposure on microhardness of Nano-composite Filtek^{TM} Z 350 XT, Nano glass ionomer Ketac N100 and Micro-hybrid composite Filtek Z250. **Materials and Methods:** 96 samples were prepared in disc shaped stainless steel molds with uniform size of (6mm) diameter and (2 mm) thickness. Samples were divided according to materials used into three groups and then each group was subdivided into subgroup according to light of curing. A single operator prepared the samples. Each subgroup was divided into two groups(eight in each) according to used remineralizing agent (GC MI Paste Plus) or not. Two curing units were used to polymerize the samples halogen Cromalux 7050 [Mega-PHYSIK GmbH & Co KG, Megadenta, Germany] and LED [Bluephase C5, IvoclarVivadent] for 40Sec. Samples were stored in a dark container in distilled water for 24hr and then one group had Vickers microhardness test and put the other one in the remineralizing agent for 7 days before microhardness test. Statistical analysis for all data were analyzed by two way analysis of ANOVA and Tukey's tests. **Results:** In all the tested materials, LED curing, whether used alone or in combination with remineralizing mouse, resulted in greater microhardness, at both the top and bottom surfaces, compared to halogen light curing alone or in combination with remineralizing mouse. Glass ionomer N100, whether cured by LED or halogen light- showed the lowest microhradness compared to Z250 and Z350 **Conclusions:** LED curing were significantly influenced the microhardness values for all tested materials. Glass ionomer showed the lowest micro-hardness compared to Z250 and Z350.

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1.Introduction

Quartz- tungsten halogen lights (QTH) are the most frequently used curing units to photo-activate resin-based dental materials, Araujo et al.,(2008); Bouillaguet et al.,(2005). Benefits include the ability to polymerize all restorative materials, irrespective of the photo-initiator added, Price et al.,(2003) . Another advantage also includes a low cost technology curing unit, Rueggeberg(1999). On the other hand, these light units develop high temperatures and have a declining power density over time due to bulb and filter aging, Daniela et al.,(2009) .

Light-emitting diode (LED) devices overcome some of the short comings of QTH LCU, Mount et al.,(2002) .These devices are composed of solid-state LEDS that use junctions of doped semiconductors based on gallium nitride to directly emit light in the blue region of the spectrum, without excessive heating, Kurachi et al.,(2001). LED curing units are very compact, promise unlimited life, Rueggeberg et al.,(2005).These curing units are very specific for the camphorquinone/ amine system, Uhl et al.,(2004).

Nanotechnology or nano-science refers to the research and development of an applied science at the atomic, molecular, or macromolecular levels, Kirk et al.,(1990) .Current composite systems also utilize nanotechnology in their development. The nano-composite is composed of nanomeric particles and nano-clusters, Davis (2003); Mitra et al.,(2003).The current study evaluated the effect of remineralization and light exposure on microhardness of nanofilled resin composite, nanofilled glass ionomer and micro-hybrid composite.

2.Materials and Methods:

2.1.Materials:

2.1.1. Remineralizing agents

Three commercial materials [Nanocomposite Filtek^{TM} Z 350 XT, Nano glass ionomer Ketac N100 and Micro-hybrid composite Filtek Z250 as a control] of shade A3 were used in this study. GC MI Paste Plus (topical crème with calcium phosphate and fluoride) was used as remineralizing agents. The commercial name, composition and manufacturer of all materials used in this study were listed in Table (1).Two light curing units Cromalux 7050 [Mega-PHYSIK GmbH & Co KG, Megadenta, Germany] at 400 m W/ cm2 and LED (Bluephase C5, IvoclarVivadent] at 400 m W/ cm2 were used in this study.

Table (1): The commercial name, the composition and manufacturer of the materials used:

Materials	Manufacture	Composition
Ketac N100 Light-Curing Nano-Ionomer Restorative	3M ESPE, St Paul, MN, USA	Deionized water, blend, including HEMA, a methacrylate-modified polyalkenoic acid. **Filler component**: methacrylatefunctional-fluoroaluminosilicate glass and nanomeres and nano-clusters.
Filtek Z350 XT Universal restorative	3M ESPE, St Paul, MN, USA	The fillers are a combination of aggregated zirconia/silica cluster filler with an average cluster particle size of 0.6 to 1.4 microns and a non-agglomerated/ non-aggregated 20 nm silica filler. The inorganic filler loading is about 78.5% by wt (59.5% by volume). It contains bis-GMA, UDMA, TEGDMA, and bis-EMA
Filtek Z250	3M ESPE, St Paul, MN, USA	This light-cured resin is filled with 60% (volume) silica/zirconia. Particle size range of 0.01-3.5 um The matrix is consisting of BIS-GMA, UDMA and Bis-EMA.
Re-mineralizing agent GC MI Paste Plus	Recaldent GC Europe N.V. Inter-leuvenlaan 13,B-3001 Leuven	CPP-ACPF,Caseinphosphopeptide-Amorphous Calcium Phosphate Fluoride. The level of fluorides 0.2%WW (900ppm)

2.2.Methods:

2.2.1.Specimens' preparation

Ninety six Samples were prepared in disc shaped stainless steel molds with uniform size of (6mm diameter and 2 mm in thickness). Samples were divided according to materials used into three groups 32 samples for each material and then each group was subdivided into subgroup according to light of curing16 samples. A single operator prepared the samples. Each subgroup was divided into two groups according for using re-mineralizing agent or not. (n=8).

Restorative material was handled according to the manufacturer's instructions. The molds were placed on flat glass plates covered with Mylar strip and then filled with restorative materials.

The material was covered with Mylar strip and a microscope slide was pressed against the mold to adapt the materials completely to the inner portion of the mold. The excess material was removed and the samples were Photo-activated for 40 sec at the top surface units. Two curing units were used to polymerize the samples halogen light and LED. Immediately after light-curing the cover slide were removed from the mold , the top surface was identified with an indelible mark and stored in the dark container in distilled water for 24hr . After that, one group had micro-hardness test and another one was put in the remineralizing agent for 7 days. Remineralizing crème changed every day and after this period, the microhardness test was performed.

2.2.2.Vickers hardness measurements

Surface hardness of the specimens was determined using Digital Display Vickers Micro-hardness Tester (Model HVS-50, LaizhouHuayin Testing Instrument Co., Ltd. China) with a Vickers diamond indenter and a 20X objective lens. A load of 200 gram was applied to the surface of the specimens for 15 seconds. Three indentations were equally placed over a circle of 1-mm diameter at the middle third of the specimens. The diagonals lengths of the indentations were measured by built in scaled micrometer and Vickers values were converted into micro-hardness values.

2.2.3. Micro-hardness calculation:.

Micro-hardness was obtained using the following equation:

$HV=1.854\ P/d^2$ .....*Where:*

HV is Vickers hardness in Kgf/mm^2,

P is the load in Kgf and

d is the average diagonals lengths in mm

2.2.4. Statistics analysis:

All data were analyzed by two way analysis of ANOVA and Tukey's post-hoc test.

3.RESULTS:

Table 2, Figure (1 and 2) showed the mean standard deviation of microhardness of the top and bottom surface of specimens of the studied materials.

Using the ANOVA test, a statistically significant difference was observed between the micro hardness of the studied materials using each of the curing techniques, whether microhardness was measured in top or bottom p<0001.

Comparing of the mean of microhardness of the three materials in top and bottom surface revealed that N100 whether cured by LED or halogen light showed the lowest microhradness compared to Z250 and Z350.

Regarding that all the tested materials, LED curing, whether used alone or in combination with remineralizing mouse, resulted in greater microhardness, both at the top and bottom surfaces, compared to halogen light curing alone or in combination with remineralizing mouse.

On the other hand, the statistical analysis revealed that remineralizing mouse improved the microhardness of top surface of Z350 and N100 cured by halogen lamp.

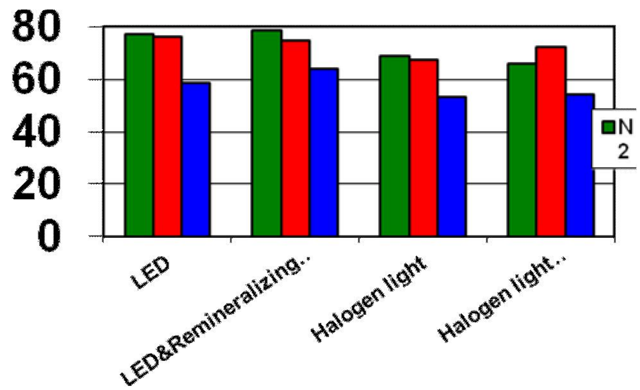

Fig. (1) Microhardness in different materials using different curing techniques at the top surface

Table (2) Mean ± standard deviation of microhardness of the top and bottom surface of specimens of the studied materials

Material	LED		LED & Remineralizing mouse		Halogen light		Halogen light & remineralizing mouse	
	Top	*Bottom*	*Top*	*Bottom*	*Top*	*Bottom*	*Top*	*Bottom*
Z250	76.938 ±0.410	71.075± 0.183	78.825± 0.198	68.643± 0.007	68.700± 0.163	68.671± 0.170	66.157± 0.14	62.175 ±0.128
Z350	76.250± 0.316	73.3± 0.214	74.938± 0.007	75.114± 0.146	67.514± 0.006	67.086± 0.008	72.171 ±0.125	65.925 ±0.008
N100	58.687± 0.247	62.012± 0.136	64.1± 0.160	57.357± 0.181	53.114± 0.008	54.271± 0.007	54.214 ±0.186	54.787 ±0.113
P value	<.0001*	<.0001*	<.0001*	<.0001*	<.0001*	<.0001*	<.0001*	<.0001*
Tukey's	for p<0.05 =0.3458	for p<0.05 =0.0590	for p<0.05 =0.0500	for p<0.05 =0.0503	for p<0.05 =0.0406	for p<0.05 =0.0423	for p<0.05 =0.0542	for p<0.05 =0.0366
	for p<0.01 =0.4712	for p<0.01 =0.0804	for p<0.01 =0.0682	for p<0.01 =0.0690	for p<0.01 =0.0557	for p<0.01 =0.0580	for p<0.01 =0.0743	for p<0.01 =0.0498

P value represents the significance of the difference between the 3 materials
*Statistically significant
Tukey's: Least significant difference (Tukey's Honestly Significant Difference)

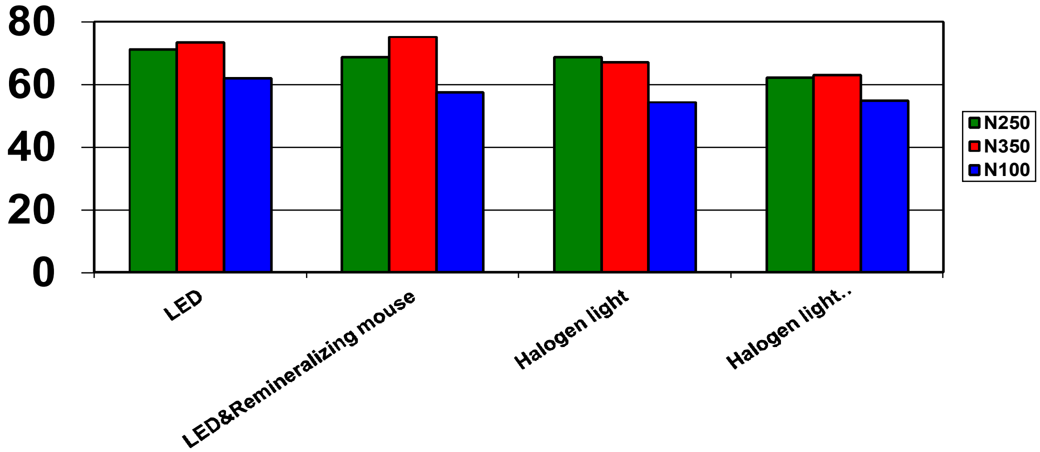

Fig. (2) Microhardness in different materials using different curing techniques at bottom surface

Using halogen light only, the microhardness of Z250 and Z350 at the top surface was greater than that of the bottom surface. In contrast, N100 showed nearest microhardness at the top and bottom surface when cured by halogen light only or halogen light and remineralizing mouse. In contrast, Z350 showed greater microhardness at the top surface when cured by halogen light and remineralizing mouse.

Discussion

The current study examined the effect of remineralization and light exposure on microhardness of nano-filled resin composite, nano-filled glass ionomer and Micro-hybrid composite.

Light-emitting diode (LED) devices overcome some of the short comings of QTH LCU , Mount et al.,(2002)

Hardness is a surface property of a material that shows its resistance against permanent deformation. Vickers hardness is a type of microhardness test which is commonly used to evaluate surface micro hardness of brittle and restorative materials , Craig and Powers (2006); Sasaki et al.,(2009).

As regards microhardness results from the current study were showed significant difference between top and bottom surfaces. This finding is in agreement with other studies that showed differences between top and bottom surface microhardness, Jandt et al.,(2000); Dunn and Bush (2002); Garcia-Godody et al.,(2004). However, there are also studies revealing no significant difference indicating the sufficient energy penetration through the material, Soh et al.,(2003) ; Okte et al.,(2005) ; RizaAlpoz et al.,(2008) .

Moreover, all the tested materials, LED curing, whether used alone or in combination with remineralizing mouse, resulted in greater microhardness, both at the top and bottom surfaces, compared to halogen light curing alone or in combination with remineralizing mouse. This may be due to higher light intensity of the newer LED devices with their narrow spectral output makes them even more efficient than conventional halogen light curing units, Rueggeberg et al.,(1993); Della Bonna et al.,(2007).This finding is in agreement with other studies that showed significant difference between the polymerization of composite resin with LED for 40s is better than that with Halogen light for 40s cured group, Rueggeberg et al.,(1993).

On the other hand glass ionomer N100 showed nearest microhardness at the top and bottom surface when cured by halogen light only or halogen light and remineralizing mouse. These may be due to two types of setting reactions take place in the light cured glass ionomer:

1) The acid base reaction between the fluoroaluminosilicate glass and the polycarboxylic acid, the same reaction as in a conventional glass ionomer, and

2)A light activated free radical polymerization of methacrylate groups of the polymer and HEMA, Young and FTIR (2002).

Since the rate of the second reaction, the photo-polymerization reaction is much faster than the first; the setting time of the cement is much shorter than that of conventional systems Rueggeberg et al.,(1993).This curing reaction gives these materials extended working time and optimal physical properties, Mount et al.,(2002).

Composite Z350 showed greater microhardness at the top surface when cured by halogen light and remineralizing mouse. The camphorquinone present in the Z350 resin composite is also found in many different brands of resin composite based dental material. Although there is an aesthetic limitation of these photoinitiator. If the polymerization process is insufficient and unreached camphorquinone remains at final restoration, Schneider et al.,(2008). Camphorquinone is chosen because of its high potential to initiate the conversion process of monomers when sensibilized with blue light, Callheiros et al.,(2008).

On the other hand, sodium fluoride (NaF) agents on ceramics, composites and sealants revealed important structural alterations that were dependent on the fluoride agent used, Cehreli et al.,(2000).Remineralizing agent had a little effect on microhardness of used restorative materials. This is may be due to effect of nanotechnology in composition of the materials.

Conclusions:

Within the limitation of this study, the following conclusions may be drawn:

1- LED curing significantly influenced the microhardness values for all tested materials.
2- Glass ionomer showed the lowest microhardness compared to Z250 and Z350.
3- Remineralizing agent had a little or no effect on microhardness of used restorative materials.

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