

DEGREE OF CONVERSION AND SURFACE HARDNESS OF TWO NANO-COMPOSITES COMPARED TO THREE OTHER TOOTH-COLORED RESTORATIVE MATERIALS

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ABSTRACT

The aim of the laboratory study was to evaluate the degree of conversion (DC) and surface hardness of two nanofilled resin composite restorative materials compared to three other tooth-colored restorative materials. A light-emitting diode curing unit was used to photo-activate a hybrid composite (Filtek™ Z250), two nanofilled composites (Tetric® EvoCeram, Filtek™ Supreme XT), a nano-ionomer (Ketac™ N100) and a compomer (Dyract® eXtra). After 24 hours dry storage in the dark at 37°C, the DC was determined using Fourier transformation infrared spectroscopy (FTIR). A microhardness tester was used to measure the Vickers Hardness Number (VHN). Data for DC and VHN were analyzed using one-way analysis of variance with Tukey's multiple comparison test, and by using Pearson's correlation test ($P=0.05$ for statistical significance). The highest mean value for DC was obtained with Tetric EvoCeram ($79.4 \pm 6.7\%$). Filtek Z250 showed a significantly lower mean DC ($58.3 \pm 1.8\%$) than any of the other tested materials ($P < 0.001$). Filtek Supreme XT, Ketac N100, Dyract eXtra and Tetric EvoCeram showed no significant DC differences among them ($P > 0.05$). Filtek Z250 had the highest VHN (97.6 ± 2.8 MPa), while the lowest value was recorded with Tetric EvoCeram (55.3 ± 2.9 MPa). The VHN was significantly different among all tested materials ($P < 0.001$). A significant and highly negative correlation existed between the DC and surface hardness ($r = 0.736$, $P < 0.01$). The study concluded that Nanofilled composites may have a higher DC and a reduced VHN compared to hybrid composites, with clinical implications. Correlation between these two properties for various nanofilled materials requires further investigation.

Key words: Nanofilled restoration, Degree of conversion, Surface hardness, Nano-ionomer

INTRODUCTION

Resin composites have been classified according to the type of filler; as hybrid, microhybrid, microfill and, more recently, nanofill.¹ Microhybrid composites have high filler loadings (more than 60% by volume), with a blend of submicron (0.04 μ m) and small particles (1-4 μ m) that provide optimal wear resistance and adequate mechanical properties.²

More recently, a newer type of light-cure resin composite (nanofilled) has been introduced to the market. Nanotechnology consists of controlling or reducing components of a material to the nanometric scale (e.g. filler sizes from 0.005-0.01 μ m) to improve the final characteristics.¹ These newer types of resin com-

posite were developed because of the increasing demand for a universal aesthetic dental material suitable for all types of direct restorations, including placement in posterior teeth.¹ Although several studies have evaluated the mechanical and physical properties of nanofilled materials^{3,4}, their long-term clinical performance, including color stability, remains to be determined.

Today, a variety of dental restorative materials available under different brand names in the market is based on nanotechnology. A recent development in the technology of resin-modified glass-ionomers is a light-cure nano-ionomer restorative material. The manufacturer of Ketac N100 (3M ESPE, St Paul, MN, USA)

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claims that the product combines the benefits of a light-cure resin-modified glass-ionomer with those of bonded nanofiller technology. The incorporation of both nano-sized fillers and nanoclusters, along with fluoroaluminosilicate glass, is claimed to result in excellent mechanical properties, high fluoride release, great aesthetics and relatively low wear.

Curing resin composite with visible light is the standard method of polymerizing resin-based materials. Blue-light-emitting diode (LED) units have been classified on the basis of their light intensities. Older LED units with an intensity of approximately 400 mW/cm² are suitable alternatives to quartz tungsten halogen (QTH) light sources. Further advancements⁵ in technology have made it possible to produce high-powered LED units with intensities of approximately 1000 mw/cm.² There is some controversy regarding different curing lights and their effects on the degree of resin monomer conversion, which is related to the physical and chemical properties of different resin-based materials. Different effects might be caused by the diversity of material components and light intensities used. In one study, second-generation LED curing units achieved significantly greater depths of cure than QTH units for all tested composites.⁶

Since predictions for the long-term durability of resin composite materials are difficult, it is important that all monomers should be effectively converted to polymers during the polymerization reaction.⁷ The degree of conversion (DC) of methacrylate bonds is the degree to which c=c is converted into c-c to form polymer chains.⁸ Fourier transformation infrared (FTIR) spectroscopy is one of the most widely used, powerful and reliable direct methods to detect the c=c stretching vibrations before and after the curing of resin materials.^{9,10} FTIR spectroscopy has the advantage of sensitivity for measuring the conversion of dental polymer chains.⁹ There are different contributing factors¹¹⁻¹³ that could influence the DC of resin composite such as the light transmission properties of the matrix¹¹, and the filler particle type, size and permeability to light.¹² Additionally, the concentration of polymerization catalyst¹³, the concentration of initiator/inhibitor in the resin¹⁴, and the type of photo-initiator system are important factors.¹⁵ The degree of conversion of a resin material may affect physical properties such as compressive strength, wear and hardness.¹⁶ Highly poly-

merized composites, characterized by increased cross-link density and low residual monomer, have been shown to exhibit greater wear resistance, hardness and flexural strength.¹⁷

Surface hardness is related to material wear resistance and the ability to maintain form stability.¹⁸ Soft surfaces are prone to damage and excessive removal during finishing and polishing procedures.¹⁹ Increasing the filler content of resin composites is associated with greater surface hardness.²⁰ The Vickers hardness test is very useful for measuring the surface hardness of brittle materials.¹⁷ Several studies have shown a good correlation between the hardness and DC.^{9, 21, 22} Conversely, one study found that polymers with the same DC exhibited different hardness numbers.¹² Further, no linear correlation was found between the DC and the microhardness of several dental resin-based composites.²³⁻²⁵

The aim of the present study was to evaluate the degree of conversion and surface hardness of selected nanofilled and other tooth-colored restorative materials. The null hypothesis tested was that, the degree of conversion and surface hardness of the tested nanofilled composites were not significantly different from the other tooth-colored restorative materials.

METHODOLOGY

Details of the five dental restorative materials evaluated in the study are shown in Table 1. The shade selected for each material was A3, and the manufacturer's instructions concerning the use of each material were carefully followed. A LED unit (Elipar™ FreeLight 2, 3M ESPE, Loughborough, UK) with an irradiance of 1000 mW/cm² was used for photo-activation. A LED radiometer (Kerr Corp., Middleton, WI, USA) was used to measure the light intensity output during specimen curing. The present study was accomplished in two phases.

A. Determinations of the Degree of Conversion (DC)

Twenty-five specimens were fabricated from the five materials (n=5 each). All of the specimens were prepared and subsequently tested by one operator (NMT). Discs (5 mm diameter x 2 mm thickness) of each tested material were prepared using a Teflon split-

TABLE 1: RESIN COMPOSITE MATERIALS TESTED

Material	Code	Manufacturer	Type	Composition	Lot no.
Tetric EvoCeram	TEC	Ivoclar Vivadent, Liechtenstein, Germany	Nano-hybrid Composite	Filler: barium glass, ytterbium trifluoride, mixed oxide 75-76 wt% or 53-55 vol% with particle size inorganic fillers 40-3000 nm, average 550 nm Matrix: dimethacrylates 17-18 wt%	K15482
Filtek Supreme XT	FXT	3M ESPE Dental Products, St Paul, MN, USA	Universal nano-filled composite	Filler: combination of aggregated zirconia/ cluster filler with particle size primarily of 5-20 nm and non-agglomerated non-aggregated of 20 nm, 78.5 wt% or 59.5 vol% Matrix: Bis-GMA, UDMA, TEGDMA, Bis-EMA resins	7LT
Filtek Z250	Z250	3M ESPE, Dental Products, St Paul, MN, USA	Hybrid composite	Filler: zirconia/silica 60 vol% with particle size 0.01-3.5 m Matrix: Bis-GMA, UDMA, Bis-EMA resins	7EG
Dyract eXtra	eX	Dentsply DeTrey GmbH, Konstanz, Germany	Polyacid-modified resin composite (Compomer)	Filler: strontium-alumino-sodium-fluoro-phosphor-silicate glass with particle size of 0.8 mm, and highly dispersed silicon dioxide Matrix: UDMA, TCB, TEGDMA, BHT, EDMA, trimethacrylate resins Strontium fluoride, iron oxide and titanium dioxide pigments	0612002660
Ketac N100	N100	3M ESPE, Dental Products, St Paul, MN, USA	Modified ionomer restorative	Filler: fluoroaluminosilicate glass, nanomer and nanoclusters average particle size 1 mm, 69 wt%. Nanofillers are discrete non-agglomerated and aggregated fillers of 5-25 nm Matrix: Vitrebond copolymer, methacrylate-modified polyalkenoic acid, HEMA	AF7AG

mould. The tip of the LED light guide was positioned on the top surface of the material for photo-activation. Irradiation time was as recommended by the manufacturer for each material (20 seconds). Each specimen was removed from the mould and stored dry in the dark at 37°C for 24 hours.

The DC was determined for each material using Fourier Transform Infra red spectrophotometer FTIR 8400S (Shimadzu, Kanda-Nishikicho, 1-chome, Chiyoda-Ku, Tokyo 101-8448 Japan). FTIR spectroscopy was completed using a potassium bromide pellet technique where un-polymerized specimens (n = 5) of each resin system were diluted in chloroform, smeared between two sodium chloride crystal discs, and then placed into a cell holder in the spectrophotometer.¹³ The concentration of methacrylate carbon-carbon bonds (c=c) was determined by obtaining a spectrum from 50 scans over a bandwidth of 1500-2000 cm⁻¹ at a resolution of 4 cm⁻¹.

Polymerized specimens (n = 5) of each resin system were also grounded with mortar and pestle into a fine powder. Subsequently, 2 mg of resin powder blended with 7 mg of Irspectropic grade (IR) potassium bromide in a specimen holder was pressed into a transparent disc using a pellet maker kit (KBr Product-A-Press, International Crystal Labs, Garfield, NJ, USA). The specimen holder was transferred to the spectrometer and a spectrum was obtained using the same parameters as for the un-polymerized specimens. A comparison was then made between the absorption peak intensities of c=c methacrylate bonds at about 1638 cm⁻¹ and the reference peak at about 1730 cm⁻¹ using a standard baseline technique before and after polymerization, according to the following formulas:²⁶

$$DC = 100 [1 - (A_p/B_p) / (A_u/B_u)]$$

A = height of c=c peak for resin

B = height of absorption peak c=O (absorption peak)

p = polymerized resin

u = un-polymerized resin

B. Determinations of the Surface Hardness

Five specimens from each of the five materials were again prepared by the same operator using a Teflon mould (8 mm diameter x 2 mm thickness) that was placed on a glass microscope slide. After insertion of the tested material according to each manufacturer’s instructions, the material was covered with a cellulose acetate sheet over which another glass slide was placed. The LED light guide tip was placed directly touching the glass slide, and curing followed each manufacturer’s recommendations (20 seconds). The specimens were ejected from the mould and stored dry as before in a light-proof container at 37°C for 24 hours.

The Vickers hardness number (VHN) was determined for each specimen, using a Micromet II microhardness tester (Buehler Ltd., Lake Bluff, IL, USA). A diamond indenter was used to apply a load of 300 gm for 20 seconds. Five values were recorded from each surface tested.

Data obtained from both tests were analyzed using the Statistical Package for the Social Sciences, version no. 13 (SPSS Corp., Chicago, IL, USA). One-way analysis of variance (ANOVA) was used to detect any statistically significant differences among the group means for DC and surface hardness, with the post hoc Tukey HSD multiple comparison test. Using the Pearson correlation test, the correlation coefficient (R²) obtained was used to evaluate the association between DC and surface hardness. The probability level for statistical significance was set at P = 0.05.

RESULTS

The DC results are presented in Table 2. One-way ANOVA found that the mean value for Tetric EvoCeram (79.4 ± 6.7%) was the highest, and that Filtek Z250 exhibited a significantly lower DC (58.3 ± 1.8%) than any of the other tested materials (P <0.001). Filtek Supreme XT, Ketac N100, Dyract eXtra and Tetric EvoCeram showed no significant differences among them (P >0.05).

The VHN results are shown in Table 3. The highest mean surface hardness value was obtained for Filtek Z250 (97.6 ± 2.8 MPa), and the lowest mean value for

TABLE 2: MEANS WITH STANDARD DEVIATIONS (SD) FOR THE DEGREE OF CONVERSIONS (DC) OF THE TESTED MATERIALS

Material (n = 5)	DC (SD) %	95% Confidence Interval for Mean	
		Lower Bound	Upper Bound
Filtek Z250	58.3 (1.8) ^a	56.0	60.5
Filtek Supreme XT	71.6 (4.4) ^b	66.2	77.0
Ketac N100	72.9 (8.7) ^b	62.1	83.7
Dyract eXtra	77.9 (4.3) ^b	72.5	83.3
Tetric EvoCeram	79.4 (6.7) ^b	71.0	87.7

Means with different superscript letters are statistically different at P<0.05

TABLE 3: MEANS WITH STANDARD DEVIATIONS (SD) FOR THE VICKER’S HARDNESS NUMBERS (VHN) OF THE TESTED MATERIALS

Material (n = 5)	VHN (SD) MPa*	95% Confidence Interval for Mean	
		Lower Bound	Upper Bound
Filtek Z250	97.6 (2.8) ^a	96.5	98.7
Filtek Supreme XT	86.9 (5.2) ^b	84.8	89.2
Ketac N100	78.4 (3.5) ^c	76.9	79.9
Dyract eXtra	66.7 (1.4) ^d	66.1	67.2
Tetric EvoCeram	55.3 (2.9) ^e	54.1	56.5

*MPa = Megapascal. Means with different superscript letters are statistically different at P<0.05

Tetric EvoCeram (55.3 ± 2.9 MPa). There were significant differences present among the means of all tested materials ($P < 0.001$).

A statistically significant negative correlation existed between the DC and VHN results ($r = -0.736$, $P < 0.01$).

DISCUSSION

The DC of Filtek Z250 in the present study recorded a significantly lower mean value than for the other tested materials. This finding might be related to the co-initiator present in Filtek Z250, as it absorbs light at a shorter wavelength than camphorquinone, and thus may not be activated by the curing unit.²⁷ On the other hand, with a high-powered LED unit, similar to that used in the present study, more photo-initiator reacts with the amine present and more free radicals are available for polymerization.²⁸ This photo-initiator is excited in the presences of light with a suitable wavelength and sufficient irradiance²⁹, which might be the contributing factor for the high DC of the other tested materials in the present study.

Another contributing factor that could influence the DC of resin composite is the filler particles. The light scattering within the resin composite might increase as the particle size of the filler approaches the wavelength of the activating light. Smaller filler particles scatter more light than larger and fewer glass particles.³⁰ This scattering reduces the amount of light that is transmitted through the resin composite. Therefore, the light scattering produced by nanofillers may interfere negatively with the physical properties of nanofilled composites.³ The larger particles of hybrid composites have a greater depth of cure as they are less affected by light scattering. However, this was not the case for Filtek Z250, and there may be some other contributing factor such as the ratio of the filler relative to the resin matrix. The higher the proportion of filler, the more difficult it is for the light to penetrate resin composite.³¹ Furthermore, in one study a flowable resin composite with less filler showed the highest DC.²¹

The DC of P60 (3M Dental Products) hybrid composite and of Filtek Supreme (3M ESPE) nanofilled composite were investigated in three studies.^{3,4,25} The DC of P60 when cured with a high-intensity light unit

(1000 mW/cm^2) was similar to that recorded for Filtek Z250 in the present study. But, by contrast to the present study finding, the DC of Filtek Supreme nanofilled composite was significantly lower than that of the P60 hybrid composite.^{3,4,25} As an explanation to the contrary finding, in the present study Filtek Supreme XT has been used, which the manufacturer stated is a replacement for Filtek Supreme, with new opacifiers added to improve the opacity of the resin composite. Opacity is a factor that can significantly affect light transmission.³²

Matrix composition also plays a role in the DC, as the methacrylate functional group is capable of copolymerization with monomer, and thus may enhance the DC. It had been suggested that amines having methacrylate functionality may be advantageous in increasing the DC in commercial systems, with an attendant increase in physical properties.²⁶ With high molecular weight monomers such as Bis-GMA there is always a significant concentration of un-reacted $\text{C}=\text{C}$ remaining within the resin when it is cured with visible light at mouth temperature. In addition to the un-reacted monomer, additional un-reacted $\text{C}=\text{C}$ structures may be present from diluents such as TEGDMA monomer or similar substances.³³ This could be the reason behind the higher DC readings of the tested nanofilled and Dyract eXtra materials. Filtek Z250 and Filtek Supreme XT have similar resin matrix compositions. But, the addition of TEGDMA to the resin matrix of Filtek Supreme XT may have resulted in a higher DC than that recorded for Filtek Z250. Cross-linking differences might be derived from the use of different monomers, and the monomer cross-linking and final polymer structure might contribute to change the resulting material's behavior.²⁴ The newer resin modified nano-ionomer Ketac N100 is also polymerized by light activation, with no redox or self curing during the initial setting.³⁵ Though having a different matrix composition, this could explain the comparable DC values found with the other tested nanofilled materials.

Several factors related to composition affect the surface hardness of restorative materials³⁵, higher filler fractions²² and larger and harder filler particles may improve some physical properties, including surface hardness.³⁶ In the present study, when compared to the nanofilled materials and Dyract eXtra, Filtek

Z250 showed the highest surface hardness, which disagreed with the findings of other investigators.^{11,37} The reason may be attributed to what was reported from previous investigations, that some materials required special unique curing energy (intensity x time) to reach the ultimate curing level.³⁸ It was also suggested that the filler is not responsible for the observed differences in properties of the tested materials. Instead, the matrix polymers³⁸ as well as their degree of cross-linking appear to have a significant effect on hardness²² which could be responsible for the study results. This could explain the different hardness values recorded among the nanofilled materials in the present study, even though they have close proportions of fillers. Variations between the present results and those reported in the literature may be attributed to different technologies, protocols, materials and storage conditions.³⁷ In the current study, specimens were stored dry, rather than wet. Storage under wet conditions could plasticize the specimen surfaces through hydrolysis, which may adversely affect surface hardness. Also, variability in the applied load by the Vickers indenter will affect hardness readings.³⁷

In the present study there was a negative correlation between the DC and the VHN surface hardness, which agreed with several investigations.^{9, 21, 22} But, other studies have found no significant correlations between the DC and hardness.^{3,4,25} Although surface hardness showed a good correlation with the DC for a specific resin, hardness values cannot be used to predict the DC when different resins are compared.⁷ Other reasons for the differing results might be related to the different protocols employed in the experiments. Raman spectroscopy was used by da Silva and others^{3,4}, and the parameters were different from those employed in the current setup. Although the measurement of an in-depth cure profile using the FTIR technique produced highly reliable results⁹, the findings of the present study and other investigations are quite varied and inconclusive. And, the properties of the nanofilled materials appear difficult to relate to specific factors because the chemistry is complex and, thus, require further investigation.

Although the nanofilled dental restorative materials tested in the present small study showed promising results, long-term controlled clinical trials are necessary to evaluate the durability of the newer materials.

CONCLUSIONS

The null hypothesis was not accepted. Under the conditions of the present study, it can be concluded that the mean degrees of conversion of a compomer and three nanofilled materials were similar and significantly higher than for a microhybrid resin composite ($P < 0.001$). However, the mean surface hardness values of all five materials were significantly different, with the hybrid composite having the highest value ($P < 0.05$). There was a strong negative correlation present between the degrees of conversion and the surface hardness values of the materials ($r = -0.736$, $P < 0.01$).

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