



An *in vitro* Evaluation of Diametral Tensile Strength and Flexural Strength of Nanocomposite vs Hybrid and Minifill Composites cured with Different Light Sources (QTH vs LED)

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ABSTRACT

Background: Composites always remained the target of discussion due to lot of controversies around it. Mechanical properties are one of them. With the introduction of new technology and emergence of various composites which combine superior strength and polish retention, nanocomposites have led to a new spark in the dentistry. A recent curing unit LED with various curing modes claims to produce higher degree of conversion.

The aim of this study was to evaluate the diametral tensile strength and flexural strength of nanocomposite, hybrid and minifill composites cured with different light sources (QTH vs LED).

Materials and methods: Seventy-two samples were prepared using different specially fabricated teflon molds, 24 samples of each composite were prepared for the diametral tensile strength (ADA specification no. 27) and the flexural strength (ISO 4049) of the 12 samples, six were cured with LED (Soft Start curing profile) and other six with QTH curing light and tested on a universal testing machine.

Results: The nanocomposite had highest diametral tensile strength and flexural strength which were equivalent to the hybrid composite and superior than the minifill composite.

Conclusion: With the combination of superior esthetics and other optimized physical properties, this novel nanocomposite system would be useful for all posterior and anterior applications.

Keywords: Diametral tensile strength, Flexural strength, Nanocomposite, Hybrid composite, Minifill composite, LED curing unit, QTH curing unit.

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INTRODUCTION

Just as you thought that the ever-ending battle for posterior restorations supremacy was beginning to die down, yet another spark to this flame is lit again! The looks of the new spark seem that this flame is sure to last a pretty long time. Now, this new spark is the 'nanocomposites'. Is not it amazing to know that world where we are living is shrinking! Today, the revolutionary development of nanotechnology has become the most highly energized disciple in science and technology. Nanotechnology also known as molecular engineering is the production of functional materials and structures in the range of 0.1 to 100 nm.^{1,2}

As most of the dental materials are quite brittle in nature and the forces acting in most clinical situations are highly distractive flexural and tensile forces, it is necessary to subject every potential dental material to stringent test for flexural and diametral tensile strength.

An ideal restoration should be functional as well esthetic. Microfills which are used for anterior restorations can offer superior polishability,¹ but fall short in posterior restorations due to lack of material strength. On the other hand, hybrids offer improved strength at the cost of esthetic quality. For long, dentists have been seeking for a universal restorative material that combines the best features of these two materials.¹⁻³ Minifills have been attempted, but none have captured both esthetics and strength in one material.¹ With the emergence of new technologies that produce nano-meter sized fillers, polymer reinforced with high percentage of nano-scale particles should show improved properties, like diametral and flexural strengths.¹

Nanocomposite uses nanotechnology in composite fabrication and is comprised of two fillers—nanoparticles and nanoclusters—which allow for higher filler loading, thus it exhibits the high strength of hybrids, in addition to the polishability of microfills.¹⁻³ But, there is lack of abundant scientific work that supports these claims.

A recent curing device LED claims to produce composites with higher degree of conversion and strength,⁹ which offers various curing regimes. ‘Soft start curing’ is one of them, which allows viscoelastic flow of the dental composite during polymerization leading to higher strength.⁹

In this study, the diametral tensile strength and flexural strength of Esthet-X (minifill) and TPH-Spectrum (hybrid) was tested against Filtek supreme—the new nanocomposite in the market which were polymerized with help of LED curing unit with soft start profile and QTH.

MATERIALS AND METHODS

Instruments used

1. Teflon mold
2. Composite placing instrument (dispodent dental product)
3. Contra-angle micromotor handpiece (NSK, Japan)
4. Fine grit diamond point (Horico, Germany)

Materials used

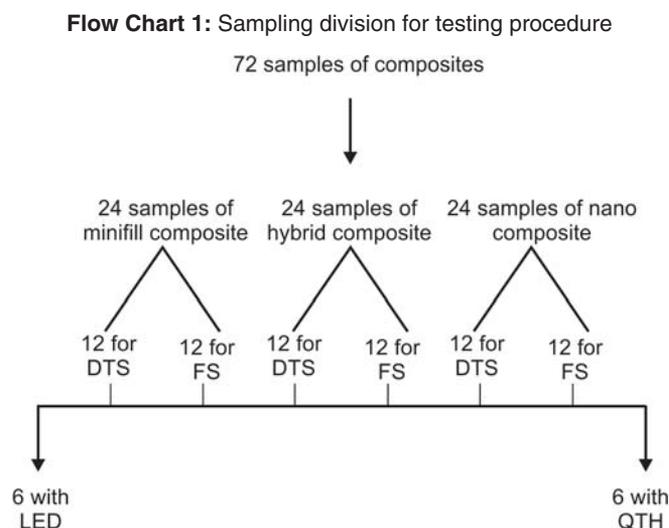
1. Filtek supreme: Nanocomposite (3M ESPE Dental Products, USA).
2. TPH spectrum: Hybrid composite (Dentsply, Germany).
3. Esthet-X: Minifill composite (Dentsply, Germany).
4. Wet Mouth—artificial saliva (ICPA products, India).

Equipments used (Figs 1 and 2)

1. Custom Fabricated Zig—diametral tensile strength (DTS), flexural strength (FS).
2. LED curing unit (3M ESPE, Elipar Free Light, USA) with inbuilt radiometer.
3. Visible light curing unit (Optilux, USA) with radiometer (confident, India).
4. Hounsfield universal testing machine (Instron, USA).

A total of 72 samples were prepared, of which 24 samples of each composite, minifill, hybrid and a nanocomposite, were prepared for diametral tensile strength (DTS) and flexural strength (FS). The samples were grouped accordingly, group1 TPH spectrum, group 2 Esthet-X and group 3 Filtek supreme.

The specimens were divided in the following manner (Flow Chart 1):



For diametral tensile strength, 12 cylindrical samples from each group with 6 mm inside diameter × 3 mm high were prepared according to modification of ADA specification no. 27.⁶ Of the 12 samples, six were cured with LED (soft start curing profile) 130 mW/cm² for 10 seconds, 370 mW/cm² for 40 seconds and other six with visible light cure of 400 mW/cm² for 40 seconds at 2 mm distance from source of light using incremental technique (Fig. 3).

For flexural strength, 12 beams from each group 25 mm length × 2 mm depth × 2 mm width were prepared according to ISO specification no. 4049 (1988).⁷ Of the 12 samples six were cured with LED (soft start curing profile) 130 mW/cm² for 10 seconds, 370 mW/cm² for 40 seconds and other six with visible light cure of 400 mW/cm² for 40 seconds at 2 mm distance from source of light using incremental technique (Fig. 4).

Before testing, the samples were stored in artificial saliva at 37°C up to 6 months and the above parameters were tested as follows by mounting them on the specialized zigs.

For flexural strength, a 3 point loading test was employed with the bar-shaped specimen being supported at the two

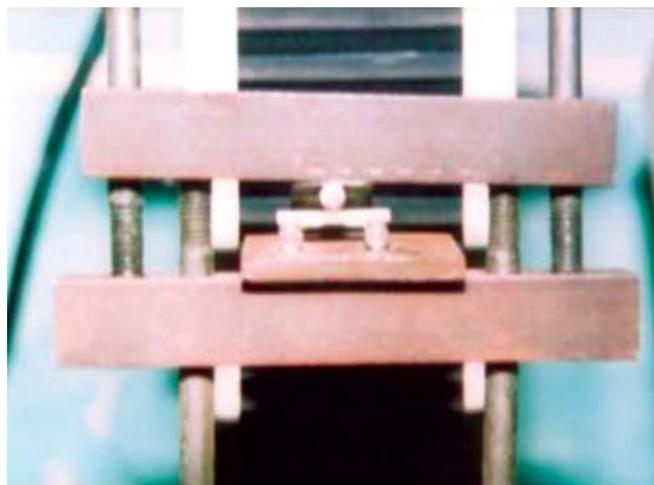


Fig. 1: Samples mounted on custom jig (FS)



Fig. 2: Samples mounted on custom jig (DTS)

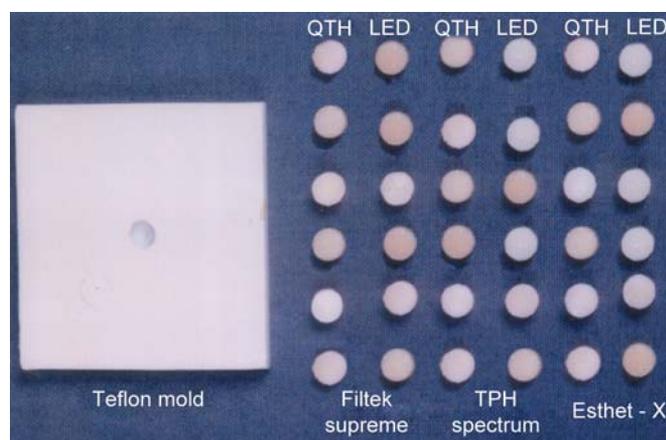


Fig. 3: Samples for diametral tensile strength

ends by the rollers set 20 mm apart. The loading was applied from the top by a central roller at the mid point of the specimen with the universal testing machine at a crosshead speed of 1 mm/min (see Fig. 1).

The flexural strength in MPa was calculated from the equation:

$$S = 3 FI / (2 Bd^2)$$

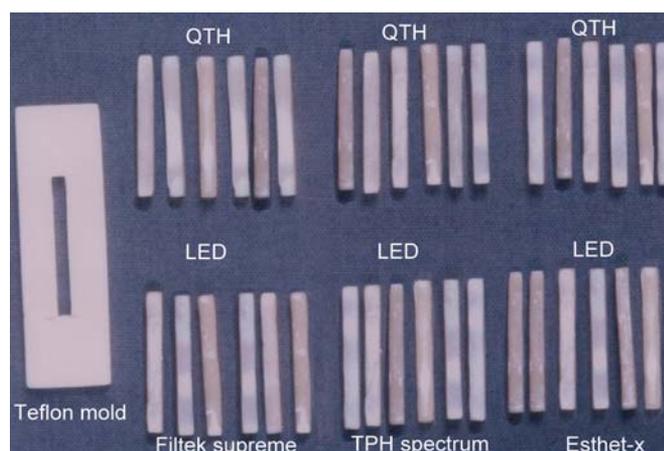


Fig. 4: Samples for flexural strength

Where F is the maximum force in N, exerted on the specimen, I is the distance in mm, between the supports, B is the breadth in mm of the specimen, and d is the depth in mm of the specimen.

For diametral tensile strength, the specimen was placed on its sides between parallel platens of the testing machine. The specimen was loaded continuously in compression at 1 cm/minute to the breaking point (see Fig. 2). The strength was computed as follows:

TS = 2 P/Dl units for TS = N/MN/M² or MPa, where TS is tensile strength, P is load at fracture (N) and D is diameter of specimen (mm) and L is Length of specimen in mm.

Statistical Analysis

Performed an analysis of variance, or ANOVA/Tukey-Kramer analysis at the 95° confidence interval or CI. p-value of 0.05 or less was considered for statistically significant difference.

RESULTS

The study was aimed to access and compare the diametral tensile strength (DTS) and flexural strength (FS) with different curing lights (LED and QTH) between the three composites used (TPH spectrum, Esthet-X and Filtek supreme).

The individual measurements mean and standard deviation values of diametral tensile strength and flexural strength of the composites cured with QTH and LED are presented in Table 1.

TPH spectrum had highest and lowest DTS values of 83.1 and 77.2 MPa, Esthet-X had values of 72.2 and 66.2 MPa and Filtek supreme had values of 86.3 and 78.6 MPa, when cured with QTH.

The DTS of composites cured with LED were as follows: TPH spectrum had highest and lowest values of 84.4, 77.4 MPa, Esthet-X obtained values of 70.6 and 65.0 MPa and Filtek supreme obtained values of 85.2 and 80.0 MPa.

The FS of composites cured with QTH were as follows: The highest and lowest values obtained with, TPH Spectrum (151.6 and 133.2 MPa), Esthet-X (147.2 and 111.8 MPa) and Filtek supreme (176.2 and 146.4 MPa).

The FS of composites cured with LED were as follows: The highest and lowest values obtained with, TPH spectrum (152.3 and 128.8 MPa), Esthet-X (146.0 and 119.9 MPa) and Filtek supreme (172.9 and 151.6 MPa).

The mean DTS values with TPH spectrum cured with QTH was 80.4 ± 2.5 MPa, Esthet-X showed lesser strength with mean value of 68.4 ± 2.5 MPa and for Filtek supreme obtained highest value 81.5 ± 2.5 MPa of all the composites tested Graph 1.

Table 1: The individual measurements, mean and standard deviation values of diametral tensile strength and flexural strength of the composites cured with QTH and LED

Sl. no.	TPH spectrum (hybrid composite)				Esthet-X (minifill composite)				Filtek supreme (nanocomposite)			
	DTS (MPa)		FS (MPa)		DTS (MPa)		FS (MPa)		DTS (MPa)		FS (MPa)	
	QTH	LED	QTH	LED	QTH	LED	QTH	LED	QTH	LED	QTH	LED
1	83.1	84.4	151.6	144.2	67.7	65.0	144.2	138.0	86.3	83.7	154.9	158.9
2	82.3	81.4	147.9	150.1	67.3	65.8	142.0	145.7	81.9	81.4	165.5	172.9
3	81.5	80.2	139.4	152.3	66.3	68.3	140.2	146.0	81.0	80.0	176.2	169.2
4	81.0	77.4	133.2	128.8	66.2	65.6	144.9	128.8	79.8	81.2	146.4	151.6
5	77.6	78.5	137.6	136.5	71.0	70.6	111.8	139.4	78.6	85.2	158.9	157.5
6	77.2	79.0	138.3	140.5	72.2	68.9	147.2	119.9	81.3	82.9	170.0	162.6
Mean	80.4	80.2	141.3	142.1	68.4	67.4	138.4	136.3	81.5	82.4	162.0	162.1
SD	2.5	2.5	6.9	8.8	2.5	2.2	13.2	10.2	2.7	1.9	10.8	7.9

ANOVA results showed DTS with QTH was different in different composites tested.

When pairwise comparisons were made by Tukey’s test, TPH spectrum and Filtek supreme showed significantly higher DTS values ($p < 0.05$) than the Esthet-X.

Samples cured with LED had the following mean DTS values—for TPH spectrum mean DTS value was 80.2 ± 2.5 MPa, Esthet-X showed lesser strength with mean value of 67.4 ± 2.2 MPa and Filtek supreme had mean value of 82.4 ± 1.9 MPa (Graph 1).

ANOVA results showed DTS with LED was different in different composites tested.

When pairwise comparisons were made by the Tukey’s test, TPH spectrum and Filtek supreme showed significantly higher DTS values ($p < 0.05$) than the Esthet-X.

For the DTS, there were no significant differences between TPH spectrum and Filtek supreme; however, there were significant differences between TPH spectrum and Esthet-X ($p < 0.05$) and between Esthet-X and Filtek supreme ($p < 0.05$), with both the curing systems employed (QTH and LED).

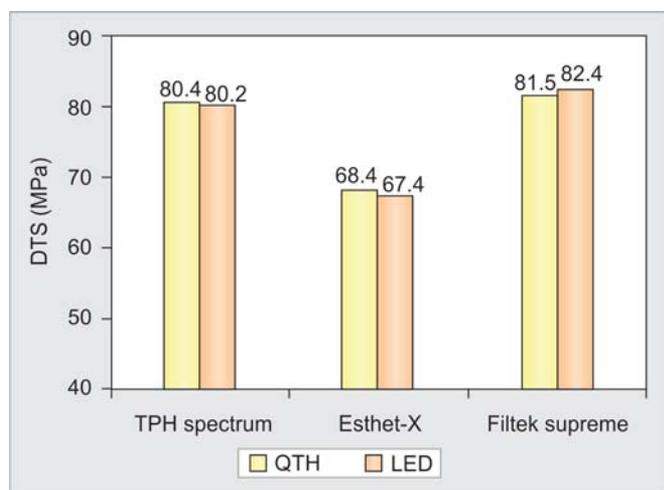
Samples cured with QTH had the following mean FS values: For TPH spectrum, mean FS value was 141.3 ± 6.9 MPa, Esthet-X showed lesser strength value of 138.4 ± 13.2 MPa and Filtek supreme had significantly higher mean value of 162.0 ± 10.8 MPa (Graph 2).

ANOVA/tukey’s post hoc test showed that the flexural strength of the Filtek supreme was higher than the TPH spectrum and Esthet-X.

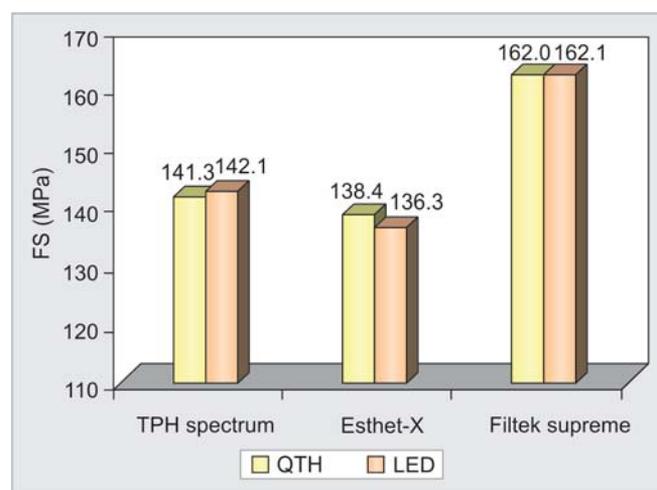
Samples cured with LED had the following mean flexural strength values: For TPH spectrum, mean FS value was 142.1 ± 8.8 MPa, Esthet-X shows lesser strength value of 136.30 ± 10.2 MPa and Filtek supreme had significantly higher mean value of 162.1 ± 7.9 MPa (see Graph 2).

ANOVA / Tukey’s post hoc test showed that the flexural strength of the Filtek supreme was higher than the TPH spectrum and Esthet-X.

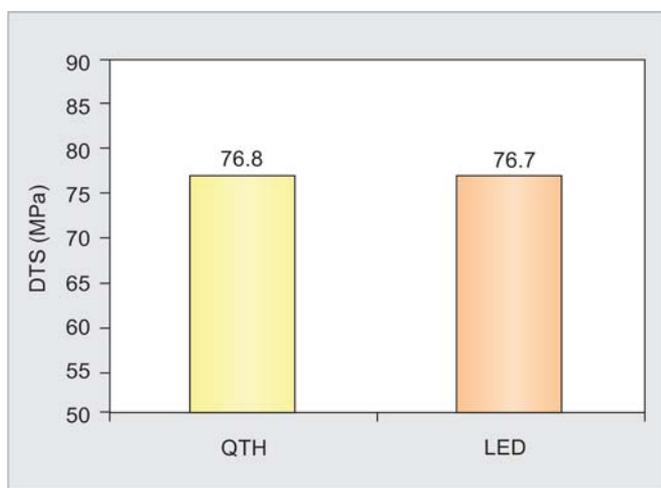
For the FS, there were no significant differences between TPH spectrum and Filtek supreme, however, there were significant differences between TPH spectrum and Filtek supreme ($p < 0.05$) and between Esthet-X and Filtek supreme ($p < 0.05$) with both the curing systems employed (QTH and LED).



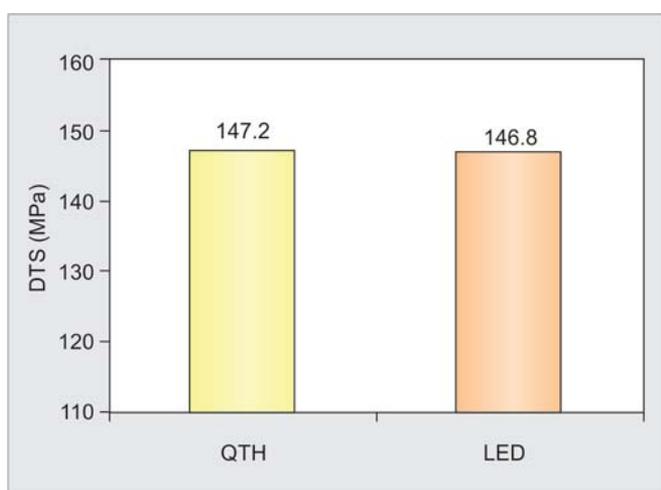
Graph 1: Mean values and comparison of diametral tensile strength of composites cured with QTH and LED



Graph 2: Mean values and comparison of flexural strength of composites cured with QTH and LED



Graph 3: Overall comparison of the effects of QTH and LED on the DTS for all the composites



Graph 4: Overall comparison of the effects of QTH and LED on the FS for all the composites

Graphs 3 and 4 list the overall comparison of the effect of QTH and LED on DTS and FS for all the composites tested.

Statistical analysis by the student's t-test showed that the mean DTS values with QTH were 76.8 ± 6.6 MPa and with LED had values of 76.7 ± 7.1 MPa for the flexural strength; the mean values with QTH were 142 ± 14.7 MPa and with LED had values of 146.8 ± 14.6 MPa.

When comparisons were made between the QTH and LED between the composites tested, there were no significant differences seen $p < 0.06$.

DISCUSSION

'There are no limits to science. Each advance merely widens the sphere of exploration' (Marconi 1874-1934).

In hybrid composites, filler consists of particles averaging $1 \mu\text{m}$ in size and offering improved strength at the cost of esthetic quality.^{1,3} On the other hand, the average

particle size of fillers in minifill composite is 0.4 to 0.6 microns. But unfortunately as these materials are abraded on being polished, the surface smoothness is reduced because of protruding filler particles and also have lower mechanical properties.¹ So, the search for strong and esthetic restorative materials continues. In the field of clinical dentistry, the latest is the nanocomposite restorative material Filtek supreme, using new technology in its development. The nanocomposite branded Filtek supreme is the recent invention and no independent research data is yet available, hence was one of main sample for our research work.

The diametral tensile strength is a property which is suited only for testing brittle materials. For this reason, the tensile strength of these materials may be considered to have more clinical relevance than the compressive strength.^{4,9}

Clinically, composite restorations can be subjected to considerable flexural stresses; for the restorations located in high stress bearing areas, high flexural strength and modulus are desired as materials with low stiffness and low modulus will deform more under masticatory stresses resulting in catastrophic failures and destruction of marginal seal between the composites and the tooth substance.⁵ So, the testing commonly employed in dental research for flexural strength, which is based on ISO 4049 is used in the present study.^{5,8}

This study was specifically designed to compare and to evaluate the recent nanocomposites, which use nano technology in composite fabrication, which is claimed to have superior mechanical properties¹⁻³ than the commonly used hybrid and the minifill composites.¹⁻³

When overall comparison was made between the composites tested cured with LED and QTH, the nanocomposite Filtek supreme had highest DTS values which were equivalent to the hybrid composite TPH spectrum and superior than the minifill composite Esthet-X, the better DTS values could be explained by the presence of higher filler load levels and the differences in the particle size.¹

For the TPH spectrum, the particle size of the inorganic fillers is 0.04 to 5 microns, the percentage of volume of total inorganic fillers is 57% and the percentage by weight is 72%.

For the Esthet-X, the average particle size is 0.7 microns, the percentage by volume of total inorganic fillers is 60% and the percentage by weight is 77.5%.

The nanocomposite Filtek supreme, contains a combination of a agglomerated /nonagglomerated 20 nm nano silica fillers and loosely bound agglomerated zirconia/silica nanoclusters, consisting of agglomerates of primary zirconia/silica particles with size of 5 to 20 nm. The cluster particle size range is 0.6 to 1.4 microns and the filler loading is 78.5% by weight.¹

The combination of nanomer-sized particles to the nanocluster formulations reduces the interstitial spacing of the filler particles. This provides for higher filler loading and better physical properties, when compared to composites containing only nanoclusters. And, also the use of spheroidal NC fillers with their broad particle distribution enables to obtain high filler loading, desirable handling characteristics and physical properties comparable to the hybrid composites.¹

When overall comparison was made between the composites tested when cured with LED and QTH, the nanocomposite Filtek supreme had highest flexural strength values than the hybrid composite TPH spectrum and the minifill composite Esthet-X, the better FS values could be explained by the presence of higher filler load and the differences in the particle size.¹

Overall the results obtained from the present study were in accordance with the previous studies.¹⁻³

When comparison was made between the QTH and LED between all the composites tested there were no significant difference seen.

The spectrum of the LED light-curing unit is focused on the 470 nm wavelength corresponding to the camphoroquinone initiator, in all the composites used in this study. At a certain light intensity, i.e. 130 mW/cm², the amount of activated starter radicals is optimal to form cross-linked long chain molecules. The results obtained with the soft cure mode are not a function of progressive increasing intensity, but a function of the irradiance.¹⁰ This could explain why at a lower intensity for the LED light curing unit the results were similar to those obtained with an halogen light curing unit.

CONCLUSION

Newly developed dental nanocomposites are expanding the armoury of restorative materials available to the dental practitioner.

With the combination of superior esthetics and other optimized physical properties, it is expected that this novel nanocomposite system would be useful for all posterior and anterior applications; and further clinical studies are needed to confirm the laboratory findings.

CLINICAL SIGNIFICANCE

The nanoparticels fillers significantly enhanced the mechanical properties of dental composites introducing a new approach to develop materials with improved properties and

thus could be used for posterior restorations as well as for anterior restorations due to their high esthetic properties.

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