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Effect of Energy Density on the Physical Properties of Resin-Based Restorative Materials when Polymerized with Quartz-Tungsten Halogen or LED-Light

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ABSTRACT

Objectives: The purpose of this study was to evaluate flexural strengths, moduli, and maximum deflection of Clearfil AP-X (APX) and Ceram-X Mono (CXM) when cured with a quartz-tungsten halogen (QTH) or an LED-light (LED).

Methods: Specimens were made according to ISO 4049 and cured with QTH or LED for 10, 20 or 60 s. Flexural strength, modulus, and deflection were determined after 24 h water storage at 37°C and after thermocycling. Statistical significance was P<.05.

Results: Flexural strength did not depend on energy density or curing light and was significantly higher for APX than for CXM but decreased after thermocycling for both materials. Modulus and deflection depended on energy density. Modulus was significantly higher for APX than for CXM and increased for APX but decreased for CXM after thermocycling. Deflection decreased with increasing energy density and decreased after thermocycling. Though energy density did not influence flexural strength, it positively correlated with flexural modulus and negatively with maximum deflection.

Conclusions: Energy density did not influence flexural strength but modulus and deflection. Thermocycling affected all material properties. The LED was as effective as the QTH. (Eur J Dent 2010;4:183-191)

Key words: Resin-based filling material; Mechanical properties; LED; Curing device; Curing time.

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INTRODUCTION

LED-curing lights are increasingly used to polymerize resin-based filling materials. These very modern curing devices offer several advantages, such as high power output and very low weight. Although the first-generation devices did not perform well,[1,2] the latest generation is reported to work optimally.[3-5] The lifetime of LEDs reaches 10,000 hours compared to approximately 50 hours for a quartz-tungsten halogen bulb. They also

cause less temperature increase during the polymerization of resin-based filling materials.[6-8]

There has been much research into the influence of LED-curing lights on the hardness,[3,9,10] shrinkage,[11] temperature rise,[3,6,8] cross-link density,[4] and degree of conversion[12-14] of resin-based filling materials. But only few studies were found considering flexural strength and flexural modulus.[9,15-17] These studies showed that the second generation of LED-curing lights performed similarly to the quartz-tungsten halogen devices (QTH). Although the energy densities of the LED-curing lights were higher than those of the QTH, significant temperature increase was not measured in the pulp chamber and increased cell damage was not observed.[3,6,8] Only one of these studies investigated flexural strength according to EN ISO 4049,[18] and none measured flexural modulus. Although some publications compared the influence of energy densities of QTH and LED on hardness[3,19-21] and compressive strength,[22] none was found that compared the influence on flexural strength, flexural modulus, and deflection. The literature has described the effect of thermocycling on the physical properties of resin-based restorative materials when cured with QTH[23,24] but has not considered LED-lights and different energy densities. Therefore, there are only few possibilities for an accurate comparison of the results. Furthermore, no literature was found about the influence of LED-curing lights on flexural properties of ormocers.

An important feature of the resin matrix is that it should absorb energy and reduce stress concentrations by providing fracture toughness or ductility to maximize damage tolerance.[25] The matrices of resin-based restorative materials were also shown to be one essential reason for brittle fracture wear.[26-28] Maximum deflection measured during a three-point-bending test was used to obtain knowledge about the elasticity or toughness, respectively, of resin materials.[29,30]

Therefore, the goal of the present investigation was to determine flexural strength (according to EN ISO 4049), flexural modulus, and deflection of an ormocer (microhybrid composite with partial silicium-organically modified resin matrix) in comparison with a microhybrid resin-based filling material when polymerized with QTH or LED-light. The null hypothesis was that there is no difference (a) between the investigated properties when irradiated with QTH or LED-light and (b) in the investigated properties between the ormocer and the microhybrid.

MATERIALS AND METHODS

The ormocer Ceram-X Mono, shade M5 (Dentsply DeTrey GmbH, Constance, Germany), and the microhybrid Clearfil AP-X, shade A3 (Kuraray Europe GmbH, Frankfurt, Germany), were used as test materials (Table 1). Ceram-X Mono shade M5 is equivalent to Clearfil AP-X shade A3. The quartz-tungsten halogen light Hilux Ultra Plus (Benlioglu Dental Inc., Ankara, Turkey) with a 10 mm light guide and the LED-light curing device SmartLite PS with an 8 mm light tip (Dentsply DeTrey GmbH, Constance, Germany) were used to polymerize the materials in the constant polymerization mode. Each time after a series of ten specimens was cured, the output of each of the curing devices was controlled with a photometer (Curing Light Meter, Benlioglu Dental Inc.). Irradiances between 750 and 850 mW/cm^2 (mean 800 ± 67 mW/cm^2) were measured for the Hilux Ultra Plus and between 1100 and 1300 mW/cm2 (mean 1200 ± 98 mW/cm^2) for the SmartLite PS. No significant decrease of the output of either device was observed. The energy density of each curing device was calculated for the different exposure times (Table 2).

The preparation of the specimens was done according to EN ISO 4049.[18] From each material, 120 specimens with a size of (25±2) mm x (2±0.1) mm x (2±0.1) mm were manufactured at 22.0 - 23.0°C (room temperature) and a relative humidity of 50%. Prior to polymerization, both sides of the specimens were covered with a 0.05 mm transparent polyester film. The initial curing location was in the center of the specimen. Two additional curing increments were used on either side of the initial curing location from the center of each specimen toward its end. The specimens were turned over, and the curing sequence was repeated on the bottom. The curing sequence resulted in a total of five curing increments on each side of each specimen (ten in total). The 120 specimens of each material were subdivided into three groups, each of 40 specimens. One half of the specimens of group 1 was cured with Hilux Ultra Plus, the other half with SmartLite PS for 10 s; the specimens of group 2 were cured for 20 s, group 3 for 60 s.

All specimens of each test material were stored for 24 h in demineralized water at 37°C in the dark. Ten specimens of each group were removed, and flexural strength and flexural modulus were investigated. The other ten specimens remained in water at 37°C for four weeks and were subsequently thermocycled 5000 times in water between water baths at +5 and +55°C prior to strength testing. The dwell time at each temperature level was 30 s, and the transit time was 15 s. To evaluate strength, the three-point-bending test was performed with a universal testing machine (Model 106.L, Test GmbH, Erkrath, Germany) at a crosshead speed of 0.75 mm min^{-1}.

Flexural strength σ was calculated in MPa by:

$$\sigma = \frac{3F \times L}{2b \times h^2}$$

Flexural modulus E was calculated by:

$$E = \frac{L^3}{4b \times h^3} \times \frac{F}{Y}$$

F = maximum strength in N
L = distance between the rests
b = width of the specimen
h = height of the specimen
F / Y = slope of linear part of the stress-strain curve

Maximum deflection was taken directly from the stress-strain-curve.

Statistical analysis

Statistical analysis was conducted with SPSS software 12.0 (SPSS Software, Munich, Germany). Means and standard deviations were calculated. Normal distribution was proven by the Kolmogoroff-Smirnoff Test. Multiple comparisons were made for each of the tested properties with the univariate Anova followed by a Scheffe post hoc test and t-tests for unpaired samples. Correlations were calculated according to Pearson. Statistical significance for all tests was considered as $P<.05$.

RESULTS

Means and standard deviations of flexural strength, flexural modulus, and maximum deflection are shown in Table 3. Significant differences of flexural strength, flexural modulus, and deflection were calculated between the microhybrid Clearfil AP-X and the ormocer Ceram-X Mono prior to and after thermocycling for all curing times and both of the curing devices (Tables 5, 6 and 7).

Clearfil AP-X showed significantly higher flexural strength than Ceram X Mono for all energy densities, curing devices, and aging conditions (Tables 3, 4 and 5). Except for Clearfil AP-X 20 s QTH-cured, neither prior to nor after thermocycling was a significant influence of energy density or curing device on flexural strength observed for the test materials (Tables 3, 4 and 5). Flexural strengths of all LED-light polymerized samples significantly decreased after thermocycling, which was not the case for all of the QTH-cured specimens (Tables 3 and 7). No correlation was found between energy density and flexural strength for any of the test materials (Tables 3 and 8).

Clearfil AP-X showed significantly higher flexural modulus than Ceram X Mono for all energy densities, curing devices, and aging conditions (Tables 3, 4 and 5). Flexural modulus increased for Clearfil AP-X with increasing curing time or energy density, respectively, and after thermocycling for both of the curing devices. The SmartLite-cured Clearfil AP-X specimens had higher modulus values than the Hilux Ultra Plus-cured specimens. In contrast to Clearfil AP-X, the flexural modulus of Ceram-X Mono remained constant or decreased after thermocycling (Tables 3, 4 and 5). Clearfil AP-X and Ceram-X Mono showed a significantly positive correlation between energy density and flexural modulus prior to as well as following thermocycling (Table 8).

Maximum deflection was significantly lower for Clearfil AP-X than for Ceram-X Mono when QTH-cured, but no differences were found for the LED-light-cured samples. No influence of curing time or curing device was detected either for non-thermocycled or for thermocyled Clearfil AP-X. Deflection decreased for Ceram-X Mono with increasing curing time and both curing devices (Tables 3 and 6). After thermocycling, the values of both test materials decreased (Tables 3 and 7). Ceram-X Mono showed a significant strong negative correlation between energy density and maximum deflection prior to and after thermocycling, but Clearfil AP-X did not. Further correlations were detected for both test materials for flexural strength and flexural modulus with maximum deflection (Table 8).

DISCUSSION

This study investigated the influence of QTH or LED-light on flexural strength, flexural modulus, and deflection of two different types of resin-based filling materials according to EN ISO 4049.[18]

Flexural strength and flexural modulus are appropriate for evaluating the quality of the light-curing process[9,15,24,31] and maximum deflection furnished some knowledge about the materials' elasticity or toughness, respectively.[29,30] Literature reported that thermocycling also had an impact on the flexural properties.[24] Since the degree of conversion not only depends on the curing conditions but also on the chemical character of the resin matrix,[32,33] a microhybrid composite (Clearfil AP-X) and a microhybrid composite with partial silicium-organically modified resin matrix, so-called ormocer, (Ceram-X Mono) were chosen for this investigation. The spectral ranges of QTH and several contemporary LED-lights (also SmartLite PS) were reported by the literature and documented in Table 2.[34,35]

Several publications have shown that the combination of energy density and exposure time has significant influence on the degree of cure, flexural strength, and flexural modulus.[31,36,37] Peutzfeldt et al[31] found higher levels of degree of cure, flexural strength, and flexural modulus for TetricCeram with increasing energy densities. They concluded that the higher the energy density, the higher the degree of cure and mechanical properties. The present study could not confirm these findings for flexural strength but could for flexural modulus,

Table 1. Test materials.

Material	Formulation	Manufacturer
Ceram-X-Mono[1] #05110000198 Shade: M5 = A3, microhybride composite with partial silicium-organically modified resin matrix (Ormocer)	Resin matrix: methacrylate modified polysiloxane, dimethacrylate resin Inorganic filler: Ba-Al-borosilicate glass, pyrogenic SiO_2 Filler load: 76 mass-%, 57 vol.-% Photoinitiator: camphorquinone Synergist: ethyl-4-diemthylamino benzoate, UV stabilizer Stabilizer: butylated hydroxy toluene	DeTrey Dentsply GmbH, Constance, Germany
Clearfil AP-X[2] #01122B, Shade: A3, microhybride	Resin matrix: Bis-GMA, Tegdma Inorganic filler: Ba-glass, silica, pyrogenic SiO_2 Filler load: 85.5 mass-%, 70 vol.-% Photoinitiator: camphorquinone Synergist: NI	Kuraray Co. Inc., Kurashiki, Japan

Bis-GMA = Bisphenol-A-dimethacrylate, Tegdma = Triethylenglycol dimetacrylate, HPMA = 3-Hydroxpropyl methacrylate, NI = No information

[1] Formulation according to the literature[3,4,39]

[2] Formulation according to the literature[1,44]

Table 2. Irradiances and energy densities of Hilux Ultra Plus and SmartLite PS.

	Curing time [s]	Irradiance [mW/cm²]	Energy density [mWs/cm²]	Spectral range [nm] *)
Hilux Ultra Plus QTH	10	800	8000	400-520, broad, Flat distribution, maximum: 520
	20	800	16000	
	60	800	48000	
SmartLite PS LED	10	1200	12000	450-470 peak: 460
	20	1200	24000	
	60	1200	72000	

Camphorquinone spectral range: 350 - 550 nm, peak: 468 nm *)

*) according to literature[22,43]

Table 3. Flexural strength, flexural modulus, maximum deflection and (standard deviation) of Clearfil AP-X and Ceram-X Mono prior to (24 h) and after thermocycling (TC).

		Clearfil AP-X				Ceram-X Mono			
		Hilux Ultra Plus (HAL)		SmartLite PS (LED)		Hilux Ultra Plus (HAL)		SmartLite PS (LED)	
		24 h	5000 TC	24 h	5000 TC	24 h	5000 TC	24 h	5000 TC
Flexural strength [MPa]	10s	114 (14)	103 (18)	124 (25)	109 (11)	86 (10)	57 (11)	87 (10)	57 (12)
	20s	148 (33)	107 (17)	122 (22)	105 (18)	69 (15)	59 (9)	79 (9)	55 (8)
	60s	125 (22)	100 (18)	131 (19)	116 (21)	74 (21)	63 (11)	86 (13)	58 (15)
Flexural modulus [MPa]	10s	9550 (330)	12400 (800)	12430 (790)	12660 (330)	6110 (350)	5730 (410)	7190 (560)	7240 (860)
	20s	11180 (640)	12780 (320)	12100 (290)	15200 (990)	6650 (850)	5560 (360)	6920 (860)	7300 (240)
	60s	12140 (490)	12560 (340)	13000 (640)	15700 (850)	6810 (640)	6750 (290)	8070 (720)	7010 (450)
Maximum deflection [mm]	10s	0.41 (0.07)	0.27 (0.03)	0.36 (0.03)	0.26 (0.03)	0.51 (0.05)	0.40 (0.03)	0.48 (0.04)	0.31 (0.03)
	20s	0.39 (0.08)	0.27 (0.04)	0.42 (0.05)	0.29 (0.03)	0.44 (0.07)	0.40 (0.03)	0.42 (0.04)	0.30 (0.05)
	60s	0.37 (0.06)	0.32 (0.03)	0.36 (0.03)	0.36 (0.05)	0.41 (0.06)	0.30 (0.04)	0.38 (0.04)	0.28 (0.06)

Table 4. Significances (bold and italic) of flexural strength and flexural modulus between the materials, curing lights and curing times after 24 hours storage in water at 37°C (P<.05).

			Flexural strength after 24 hours in water at 37°C											
			Hilux Ultra Plus						SmartLite PS					
			Clearfil AP-X			Ceram-X Mono			Clearfil AP-X			Ceram-X Mono		
			10s	20s	60s	10s	20s	60s	10s	20s	60s	10s	20s	60s
Hilux Ultra Plus	Clearfil AP-X	10s		0.253	0.999	0.402	***0.005***	***0.028***	1.000	1.000	0.960	0.499	***0.001***	0.405
		20s	***0.002***		0.841	***0.000***	***0.000***	***0.000***	0.794	0.681	0.980	***0.000***	***0.000***	***0.000***
		60s	***0.000***	0.497		***0.031***	***0.000***	***0.000***	1.000	1.000	1.000	***0.049***	***0.003***	***0.031***
	Ceram-X Mono	10s	***0.000***	***0.000***	***0.000***		0.965	0.999	***0.043***	0.079	***0.004***	1.000	1.000	1.000
		20s	***0.000***	***0.000***	***0.000***	0.975		1.000	***0.000***	***0.000***	***0.000***	0.934	1.000	0.964
		60s	***0.000***	***0.000***	***0.000***	0.875	1.000		***0.001***	***0.002***	***0.000***	0.996	1.000	0.999
Smart Lite PS	Clearfil AP-X	10s	***0.000***	0.081	1.000	***0.000***	***0.000***	***0.000***		1.000	1.000	0.066	***0.004***	***0.044***
		20s	***0.000***	0.600	1.000	***0.000***	***0.000***	***0.000***	0.999		1.000	0.117	***0.010***	0.080
		60s	***0.000***	***0.000***	0.667	***0.000***	***0.000***	***0.000***	0.971	0.481		***0.007***	***0.000***	***0.004***
	Ceram-X Mono	10s	***0.000***	***0.000***	***0.000***	0.203	0.974	0.999	***0.000***	***0.000***	***0.000***		1.000	1.000
		20s	***0.000***	***0.000***	***0.000***	0.671	1.000	1.000	***0.000***	***0.000***	***0.000***	1.000		1.000
		60s	***0.000***	***0.000***	***0.000***	***0.000***	***0.013***	0.071	***0.000***	***0.000***	***0.000***	0.546	0.131	
			Flexural modulus after 24 hours in water at 37°C											

which was detected to correlate strongly positively with energy density for both test materials prior to and after thermocycling (Table 8). However, due to the restricted number of experimental groups, the present study might have failed to reveal a significant influence of the combination of energy density and curing time on flexural strength.

The correlation of energy density and flexural modulus found in the present investigation was positive and very strong prior to and after thermo-

Table 5. Significances (bold and italic) of flexural strength and flexural modulus between the materials, curing lights and curing times after 30 days storage in water at 37°C followed by 5000 thermocycles between + 5 and + 55°C (P<.05).

			Flexural strength after 24 hours in water at 37°C followed by 5000 thermocycles											
			Hilux Ultra Plus						SmartLite PS					
			Clearfil AP-X			Ceram-X Mono			Clearfil AP-X			Ceram-X Mono		
			10s	20s	60s	10s	20s	60s	10s	20s	60s	10s	20s	60s
Hilux Ultra Plus	Clearfil AP-X	10s		1.000	1.000	***0.000***	***0.000***	***0.000***	1.000	1.000	0.967	***0.000***	***0.000***	***0.000***
		20s	0.998		1.000	***0.000***	***0.000***	***0.000***	1.000	1.000	0.999	***0.000***	***0.000***	***0.000***
		60s	1.000	1.000		***0.000***	***0.000***	***0.002***	0.999	1.000	0.869	***0.000***	***0.000***	***0.000***
	Ceram-X Mono	10s	***0.000***	***0.000***	***0.000***		1.000	1.000	***0.000***	***0.000***	***0.000***	1.000	1.000	1.000
		20s	***0.000***	***0.000***	***0.000***	1.000		1.000	***0.000***	***0.000***	***0.000***	1.000	1.000	1.000
		60s	***0.000***	***0.000***	***0.000***	0.156	0.037		***0.000***	***0.000***	***0.000***	1.000	1.000	1.000
Smart Lite PS	Clearfil AP-X	10s	1.000	1.000	1.000	***0.000***	***0.000***	***0.000***		1.000	1.000	***0.000***	***0.000***	***0.000***
		20s	***0.000***	***0.000***	***0.000***	***0.000***	***0.000***	***0.000***	***0.000***		0.993	***0.000***	***0.000***	***0.000***
		60s	***0.000***	***0.000***	***0.000***	***0.000***	***0.000***	***0.000***	***0.000***	0.983		***0.000***	***0.000***	***0.000***
	Ceram-X Mono	10s	***0.000***	***0.000***	***0.000***	***0.001***	***0.000***	0.979	***0.000***	***0.000***	***0.000***		1.000	1.000
		20s	***0.000***	***0.000***	***0.000***	***0.000***	***0.000***	0.941	***0.000***	***0.000***	***0.000***	1.000		1.000
		60s	***0.000***	***0.000***	***0.000***	***0.014***	***0.002***	1.000	***0.000***	***0.000***	***0.000***	1.000	1.000	
			Flexural modulus after 24 hours in water at 37°C followed by 5000 thermocycles											

Table 6. Significances (bold and italic) of maximum deflection prior to and after thermocycling between the materials, curing lights and curing times (P<.05).

			Maximum deflection after 24 hours in water at 37°C											
			Hilux Ultra Plus						SmartLite PS					
			Clearfil AP-X			Ceram-X Mono			Clearfil AP-X			Ceram-X Mono		
			10s	20s	60s	10s	20s	60s	10s	20s	60s	10s	20s	60s
Hilux Ultra Plus	Clearfil AP-X	10s		1.000	0.998	0.145	1.000	1.000	0.988	1.000	0.937	0.691	1.000	1.000
		20s	0.889		1.000	***0.039***	0.983	1.000	1.000	1.000	1.000	0.338	0.999	1.000
		60s	0.883	1.000		***0.006***	0.879	1.000	1.000	0.992	1.000	0.114	0.997	1.000
	Ceram-X Mono	10s	0.877	***0.041***	***0.039***		0.694	0.098	***0.003***	0.275	0.001	***1.000***	0.383	***0.020***
		20s	***0.044***	***0.000***	***0.000***	0.943		0.999	0.762	1.000	0.600	0.987	1.000	0.965
		60s	1.000	0.993	0.993	0.611	***0.011***		0.998	1.000	0.992	0.569	1.000	1.000
Smart Lite PS	Clearfil AP-X	10s	1.000	0.869	0.863	0.930	0.080	1.000		0.970	1.000	0.060	0.931	1.000
		20s	0.736	1.000	1.000	***0.016***	***0.000***	0.964	0.710		0.916	0.841	1.000	0.999
		60s	1.000	0.999	0.999	0.469	***0.005***	1.000	1.000	0.988		***0.024***	0.842	1.000
	Ceram-X Mono	10s	1.000	0.965	0.963	0.792	***0.029***	1.000	1.000	0.884	1.000		0.914	0.997
		20s	1.000	0.998	0.938	0.497	***0.006***	1.000	1.000	0.985	1.000	0.939		1.000
		60s	0.994	1.000	1.000	0.185	***0.001***	1.000	0.990	1.000	1.000	0.999	1.000	
			Maximum deflection after 24 hours in water at 37°C followed by 5000 thermocycles											

cycling for both of the test materials. The lower correlation value for Ceram-X Mono after TC (0.35, P<.0000) might indicate that the flexural modulus did not increase linearly with energy density, as was determined in the study by Peutzfeldt et al.[31] Another explanation might be that the modulus decreased significantly after thermocycling. A certain explanation was not possible because of the limitations of this study. The results also show (Tables 2 and 3) that the highest energy density (SmartLite PS, 60 s curing time) resulted in the highest flexural modulus for both of the test materials. These results strongly supported the existing literature.

Energy density correlated strongly negatively with maximum deflection for Ceram-X Mono, indicating a higher degree of cure (Table 8), and was thus also in accordance with the results of Peutzfeldt et al.[31] The fact that no correlation between energy density and deflection was found for Clearfil AP-X (Table 8) might be explained by the higher filler / matrix ratio that over-compensated the influence of energy density on deflection but not on flexural modulus, since this effect was much stronger.

The findings of the present study also showed that the formulation of the material itself influenced flexural strength, flexural modulus, and deflection (Tables 3 to 6). The difference in flexural strength was not only caused by the higher filler content[38] of Clearfil AP-X but also by the filler type (agglomerated pyrogenic SiO_2) and the high con-

Table 7. Significances (bold and italic) between the 24 h storage in water at 37°C and the 30 days storage in water at 37°C followed by 5000 thermocycles between +5 and +55°C (P<.05).

			Flexural strength	Flexural modulus	Max. deflection
Hilux Ultra Plus	Clearfil AP-X	10s	0.124	***0.000***	***0.000***
		20s	***0.000***	***0.000***	***0.000***
		60s	***0.001***	0.057	***0.015***
	Ceram-X Mono	10s	***0.000***	0.150	***0.000***
		20s	0.181	***0.000***	0.099
		60s	0.125	0.830	***0.000***
Smart Lite PS	Clearfil AP-X	10s	***0.039***	0.396	***0.000***
		20s	***0.024***	***0.000***	***0.000***
		60s	***0.038***	***0.000***	0.980
	Ceram-X Mono	10s	***0.000***	0.855	***0.000***
		20s	***0.002***	0.156	***0.000***
		60s	***0.000***	***0.000***	***0.000***

Table 8. Correlations of light dose with flexural modulus and maximum deflection as wall as of maximum deflection with flexural strength and modulus after 24 h storage (24 h) and after thermocycling (TC) (P<.05).

	Correlation of energy density with			
	Flexural modulus		Maximum deflection	
	24 h	TC	24 h	TC
Clearfil AP-X	0.593 (P<.000)	0.579 (P<.000)	none	none
Ceram-X Mono	0.528 (P<.000)	0.349 (P=0.007)	-0.591 (P<.000)	-0.435 (P<.000)
	Correlation of maximum deflection with			
	Flexural modulus		Flexural strength	
	24 h	TC	24 h	TC
Clearfil AP-X	-0.411 (P=0.005)	none	none	0.713 (P<.000)
Ceram-X Mono	-0.397 (P=0.006)	-0.607 (P<.000)	0.447 (P=0.001)	0.556 (P<.000)

tent of the more rigid Bis-GMA-containing organic matrix. Ceram-X Mono's organic matrix, containing methacrylate modified polysiloxanes and finely dispersed SiO_2 particles, was more elastic.[4,39-41] Clearfil AP-X was also found to have a significantly higher flexural modulus than Ceram-X Mono mainly due to its higher filler content (Table 1). The positive correlation between filler content, flexural strength, or flexural modulus was previously reported by Rodrigues Junior et al.[38] The strong negative correlation of flexural modulus with maximum deflection showed the loss of elasticity with increasing flexural modulus or increasing filler content, respectively.

EN ISO 4049[18] requires flexural strength ≥ 80 MPa, and the literature recommends flexural modulus ≥ 10000 MPa for resin-based filling materials used in occlusal areas.[24] Only Clearfil AP-X fulfilled these requirements prior to and after thermocycling independent of the light-curing device. The results showed that there were no significant differences between the flexural strength values of Ceram-X Mono and only for the 20 s irradiated samples of Clearfil AP-X when cured with QTH or LED-light. Both of the materials behave rather similarly after thermocycling independent of the curing light - sometimes flexural strength decreased and sometimes it did not. No correlation with the curing device was found. These findings supported the literature,[9,14,16,42] which concluded that LED-lights were as effective as QTH for polymerization of the materials used.

Furthermore, it was found that the flexural modulus of Clearfil AP-X remained constant or even increased after thermocycling, whereas the modulus of Ceram-X Mono remained constant or even decreased. Such behaviour of microhybrids and ormocers was also reported in the literature, and it was concluded that the significantly lower filler content of the ormocer could be one possible cause.[24] The test materials of this study also differed significantly in filler content, so that the same conclusion might be drawn. Finally, as already discussed in a preceding paragraph, LED-lights providing high energy densities resulted in significantly higher flexural moduli.

CONCLUSIONS

Energy density did not influence flexural strength but did influence modulus and deflection. The thermocycling process affected all tested properties of the materials. The LED was as effective as the QTH for polymerization of the materials used. Therefore, part (a) of the null hypothesis is accepted for flexural strength, rejected for flexural modulus, and partially rejected for maximum deflection, and part (b) was rejected.

REFERENCES

1. Leonard D, Charlton D, Roberts H, Cohen M. Polymerization efficiency of LED curing lights. *J Esthet Restor Dent* 2002;14:286-295.
2. Uhl A, Mills RW, Vowles RW, Jandt KD. Knoop hardness depth profiles and compressive strength of selected dental composites polymerized with halogen and LED light curing technologies. *J Biomed Mater Res* 2002;63:729-738.
3. Schneider LF, Consani S, Correr-Sobrinho L, Correr AB, Sinhoreti MA. Halogen and LED light curing of composite: temperature increase and Knoop hardness. *Clin Oral Investig* 2006;10:66-71.
4. Yap AU, Soh MS, Han TT, Siow KS. Influence of curing lights and modes on cross-link density of dental composites. *Oper Dent* 2004;29:410-415.
5. Tsai PC, Meyers IA, Walsh LJ. Depth of cure and surface microhardness of composite resin cured with blue LED curing lights. *Dent Mater* 2004;20:364-369.
6. Knezevic A, Tarle Z, Meniga A, Sutalo J, Pichler G. Influence of light intensity from different curing units upon composite temperature rise. *J Oral Rehabil* 2005;32:362-367.
7. Knezevic A, Tarle Z, Meniga A, Sutalo J, Pichler G, Ristic M. Degree of conversion and temperature rise during polymerization of composite resin samples with blue diodes. *J Oral Rehabil* 2001;28:586-591.
8. Uhl A, Volpel A, Sigusch BW. Influence of heat from light curing units and dental composite polymerization on cells in vitro. *J Dent* 2006;34:298-306.
9. Campregher UB, Samuel SM, Fortes CB, Medina AD, Collares FM, Ogliari FA. Effectiveness of second-generation light-emitting diode (LED) light curing units. *J Contemp Dent Pract* 2007;8:35-42.
10. Hasler C, Zimmerli B, Lussi A. Curing capability of halogen and LED light curing units in deep class II cavities in extracted human molars. *Oper Dent* 2006;31:354-363.
11. Uhl A, Mills RW, Rzanny AE, Jandt KD. Time dependence of composite shrinkage using halogen and LED light curing. *Dent Mater* 2005;21:278-286.
12. Bala O, Olmez A, Kalayci S. Effect of LED and halogen light curing on polymerization of resin-based composites. *J Oral Rehabil* 2005;32:134-140.

13. Fleming GJ, Khan S, Afzal O, Palin WM, Burke FJ. Investigation of polymerisation shrinkage strain, associated cuspal movement and microleakage of MOD cavities restored incrementally with resin-based composite using an LED light curing unit. *J Dent* 2007;35:97-103.
14. Soh MS, Yap AU, Yu T, Shen ZX. Analysis of degree of conversion of LED and halogen lights using micro-Raman spectroscopy. *Oper Dent* 2004;29:571-577.
15. Beun S, Glorieux T, Devaux J, Vreven J, Leloup G. Characterization of nanofilled compared to universal and microfilled composites. *Dent Mater* 2007;23:51-59.
16. Stahl F, Ashworth SH, Jandt KD, Mills RW. Light-emitting diode (LED) polymerisation of dental composites: flexural properties and polymerisation potential. *Biomaterials* 2000;21:1379-1385.
17. Besnault C, Pradelle-Plasse N, Picard B, Colon P. Effect of a LED versus halogen light cure polymerization on the curing characteristics of three composite resins. *Am J Dent* 2003;16:323-328.
18. EN ISO 4049:2000 Dentistry - Polymer-based filling, restorative and luting materials.
19. Price RB, Felix CA. Effect of delivering light in specific narrow bandwidths from 394 to 515nm on the micro-hardness of resin composites. *Dent Mater* 2009;25:899-908.
20. Mobarak E, Elsayad I, Ibrahim M, El-Badrawy W. Effect of LED light-curing on the relative hardness of tooth-colored restorative materials. *Oper Dent* 2009;34:65-71.
21. Gritsch K, Souvannasot S, Schembri C, Farge P, Grosgogeat B. Influence of light energy and power density on the microhardness of two nanohybrid composites. *Eur J Oral Sci* 2008;116:77-82.
22. Silva CM, Dias KR. Compressive strength of esthetic restorative materials polymerized with quartz-tungsten-halogen light and blue LED. *Braz Dent J* 2009;20:54-57.
23. Kwon YH, Jeon GH, Jang CM, Seol HJ, Kim HI. Evaluation of polymerization of light-curing hybrid composite resins. *J Biomed Mater Res B Appl Biomater* 2006;76:106-113.
24. Janda R, Roulet JF, Latta M, Ruttermann S. The effects of thermocycling on the flexural strength and flexural modulus of modern resin-based filling materials. *Dent Mater* 2006;22:1103-1108.
25. Pilato LA, Michno MJ. Advanced composite materials. Editor: Springer, New York, 1994.
26. Sambasivan S, Fischer DA, Shen MC, Hsu SM. Molecular orientation of ultrahigh molecular weight polyethylene induced by various sliding motions. *J Biomed Mater Res B Appl Biomater* 2004;70:278-285.
27. Edidin AA, Jewett CW, Kalinowski A, Kwarteng K, Kurtz SM. Degradation of mechanical behavior in UHMWPE after natural and accelerated aging. *Biomaterials* 2000;21:1451-1460.
28. Teoh SH, Martin RL, Lim SC, Lee KH, Mok CK, Kwok WC. Delrin as an occluder material. *ASAIO Trans* 1990;36:M417-421.
29. Kostoulas I, Kavoura VT, Frangou MJ, Polyzois GL. Fracture force, deflection, and toughness of acrylic denture repairs involving glass fiber reinforcement. *J Prosthodont* 2008;17:257-261.
30. Uzun G, Keyf F. The effect of fiber reinforcement type and water storage on strength properties of a provisional fixed partial denture resin. *J Biomater Appl* 2003;17:277-286.
31. Peutzfeldt A, Asmussen E. Resin composite properties and energy density of light cure. *J Dent Res* 2005;84:659-662.
32. Sideridou I, Tserki V, Papanastasiou G. Effect of chemical structure on degree of conversion in light-cured dimethacrylate-based dental resins. *Biomaterials* 2002;23:1819-1829.
33. Peutzfeldt A. Resin composites in dentistry: the monomer systems. *Eur J Oral Sci* 1997;105:97-116.
34. Owens BM, Rodriguez KH. Radiometric and spectrophotometric analysis of third generation light-emitting diode (LED) light-curing units. *J Contemp Dent Pract* 2007;8:43-51.
35. Wendl B, Droschl H, Kern W. A comparative study of polymerization lamps to determine the degree of cure of composites using infrared spectroscopy. *Eur J Orthod* 2004;26:545-551.
36. Asmussen E, Peutzfeldt A. Flexural strength and modulus of a step-cured resin composite. *Acta Odontol Scand* 2004;62:87-90.
37. Musanje L, Darvell BW. Polymerization of resin composite restorative materials: exposure reciprocity. *Dent Mater* 2003;19:531-541.
38. Rodrigues Junior SA, Zanchi CH, Carvalho RV, Demarco FF. Flexural strength and modulus of elasticity of different types of resin-based composites. *Braz Oral Res* 2007;21:16-21.
39. Turssi CP, Ferracane JL, Ferracane LL. Wear and fatigue behavior of nano-structured dental resin composites. *J Biomed Mater Res B Appl Biomater* 2006;78:196-203.
40. Scientific Documentation Ceram-X Mono, DeTrey Dentsply GmbH, Constance, Germany, 2003.
41. Instructions for Use Ceram-X Mono, DeTrey Dentsply GmbH, Constance, Germany, 2003.
42. Soh MS, Yap AU. Influence of curing modes on crosslink density in polymer structures. *J Dent* 2004;32:321-326.