

INFLUENCE OF THE PHOTOACTIVATION MODE AND THE DISTANCE OF LIGHT-CURING UNIT IN MECHANICAL PROPERTIES OF SILORANE AND METHACRYLATE BASED RESINS

ABSTRACT

OBJECTIVES: This in vitro work had as aim evaluate the effect of photoactivation mode and the distance of light-curing unit (LCU) in microhardness Knoop (KHN) and the diametrical tensile strength (DTS) of methacrylate and silorane-based resins. MATERIALS AND METHODS: Filtek Z250 (methacrylate-based resin) and Filtek P90 (silorane-based resin), both from 3M Espe, were selected for this work. The photoactivation were performed by one of the following modes: Valo (Ultradent) at 1000 mW/cm2 X 18 s (S); 1400 mW/cm2 X 12 s (HP); 3200 mW/cm2 X 6 s (PE); and XL 3000 (3M Espe) at 450 mW/cm2 X 40 s (XL). Resin composite were inserted in one increment into a bipartide Teflon matrix (5mm X 2mm) and photoactivated at 0mm, 3 or 6mm from the increment surface, according to the experimental groups. After the confection, the specimens (n=5) were submitted to KHN on the top (T) and on the bottom (B), and to DTS. Data were analyzed through ANOVA/Tukey tests (α =5%). **RESULTS:** It was observed that Filtek Z250 presented values of KHN equal or higher than Filtek P90. The surface T presented higher values of KHN than B. For both composite resins, the values of KHN on the surface B were lower, as higher the distance of LCU. In relation to DTS, the higher values were observed in Filtek Z250. CONCLUSION: Silorane base composite resin presented lower mechanical properties when compared to the methacrylate base resin. The distance of LCU is able to influence the microhardness of bottom surface.

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KEYWORDS

Composite resin. Silorane. Light-curing source. Polymerization.
Microhardness. Diametral tensile.

INTRODUCTION

From their introduction in the market, composite resins have become popular and object of several researches which aimed at their improvement.¹ Nowadays, they have been the material of choice, not only for anterior restorations, but also for posterior ones, because the aesthetic is increasingly required by society, and due to the considerable improvements on the properties of composites, what provide better durability of adhesive procedures in direct restorations.²

However, even this material had been improved; important limitations remain, mainly related to the polymerization shrinkage.³ Some alternatives have been proposed to minimize the stress related to polymerization shrinkage, such the use of incremental insertion technique of the material, modulation of photoactivation, use of interlayer of low viscosity composites, and recently through changes in the composite resins formulation.⁴

A recent alternative in the chemical formulation of the material is a substitution of methacrylate monomer by chemical combination among siloxane and oxirane components, where the reaction of polymerization is based on the opening of cationic rings of oxirane radicals (responsible by low shrinkage), while siloxane gives hydrophobic nature to the material.⁵

In view of this recent development, the aim of this study was to evaluate the influence of the photoactivation mode and the distance of light-curing unit on the microhardness and diametrical tensile strength of methacrylate and silorane-based resins.

MATERIAL AND METHODS

To perform this study, methacrylate-based resin Filtek Z250 and silorane-based resin Filtek P90 were used, both from 3M ESPE (Chart 1).

Chart 1: Description of composite resins used in this study.

Composite Resin (shade)	/ Composition				
Manufacturer					
Filtek Z250 (shade A2)	Inorganic phase: 60 vol%,				
3M ESPE, St. Paul, MN,	silica and zirconia particles				
USA	(0.01-3.5 μm).				
Organic matrix: Bis-GMA, Bis-					
	EMA, and UDMA.				
Filtek P90 (shade A2)	Inorganic phase: 55 vol%,				
3M ESPE, St. Paul, MN,	quartz and yttrium fluoride				
USA	particles (0.04-1.7 μm).				
	Organic matrix: Silorane.				

Specimens:

For the manufacture of specimens, a circular Teflon matrix containing a cylindrical cavity in the center with 5mm of diameter and 2mm of thickness was used. A polyester strip was positioned underneath the Teflon matrix and the composite resin was inserted in a single increment, with enough volume to allow a small excess of material, and another polyester strip and a glass slide on this excess of material. After the positioning of the glass slide, a digital pressure was

performed by 10 seconds, in order to remove the excess of material and let the composite surface on the level. The glass slider was removed and the polyester strip remained in the same position. Two different light-curing units (LCUs) were selected for this study: third generation LED LCU (Valo-Ultradent) and quartz-tungsten-halogen LCU (XL 3000 - 3M Espe). The specimens were photoactivated in three different distances (0mm, 3 and 6 mm) from the tip-curing to the specimens, according to the experimental groups, using one of the following photoactivation modes: 1. XL3000 (XL): 450 mW/cm² during 40 s (18 J/cm²); 2. VALO STANDARD (S): 1000 mW/cm² during 18 s (18 J/ cm²); 3. VALO HIGH POWER (HP): 1400 mW/cm² during 3 cycles of 4 s (16.8 J/cm²); 4. VALO PLASMA EMULATION (PE): 3200 mW/cm² during 2 cycles of 3 s (19.2 J/cm²).

Microhardness Knoop Test (n=5):

After the confection, the specimens were taken to the microdurometer HMV Shimadzu to evaluate the microhardness Knoop. The indentations were carried out on the top and bottom surfaces, in 5 points; the load of 10g was applied during 10 seconds.

For each surface, it was calculated the average of 5 indentations, which were transformed in numbers of Hardness Knoop (KHN – Knoop Hardness Number), using the following formula: $KHN = L/I^2$.CP, where L correspond to the load applied, I the higher diagonal of penetration and CP the constant of projected area 14229.

Diametrical Tensile Strength Test (n=5):

After the microhardness test, the specimens were taken to the Universal Test Machine Instron model 4411 (Instron Inc. Canton, MA, EUA) in an apparatus to the diametrical tensile strength test. DTS test was performed at 0.5 mm/min, until occur the fracture of specimen. The values obtained were inserted in the following formula: $R = 2 L/\pi.D.h$, where R = resistance; L = load, D = diameter, h = height.

RESULTS

Microhardness Knoop:

The results of microhardness Knoop and the standard deviation are presented in Table 1. Methacrylate-based composite resin presented higher values of KHN than silorane-based composite resin in all the experimental conditions evaluated. For both materials, the top surface presented higher microhardness values than the bottom surface. In general way, both for methacrylate and silorane-base resin, the increase in the distance of light curing source resulted in a decrease of microhardness in the bottom surface, and the lower values on the bottom surface were obtained with distance of 6 mm and the higher ones at 0mm.

Diametrical Tensile Strength:

The results of diametric tensile strength and standard deviations are presented in table 2. Methacrylate-based resin Filtek Z250 presented higher values DTS than silorane-based resin Filtek P90. Independent on the

distance of LCU and for both materials, the higher results of DTS were obtained with XL source and the lower ones with S source, with significant statistical difference between them and they were not different from others.

Table 1. Results of microhardness Knoop (standard deviation).

T250 P90	Composite resin		Distance	Photoactivation mode Surface		
3	0	P90	Z250			
Bottom 6 *38.81(11.62) Aa *25.51(5.08) Bottom 0 36.41(10.56) Aa 21.78(4.9) 3 33.27(8.89) Aa 18.81(9.19) 6 30.13(14.22) Aa 14.8(2.82) S Top 0 *37.11(7.93) Aa *31.18(2.13) 3 *37.39(11.34) Aa *34.23(5.11) 6 *36.07(6.8) Aa *33.64(8.87) Bottom 0 30.15(4.3) Aa 15.82(7.88) 3 35.28(11.19) Aab 20.88(5.3) A 4 20.49(10.28) Ab 21.68(5.81) HP Top 0 *38.56(5.98) Aa *36.79(2.28) 3 *37.28(4.17) Aa *27.15(4.53) 6 *35(4.87) Aa *33.59(5.38) Bottom 0 29.92(1.96) Aa 27.38(7.26) 3 29.64(1.93) Aab 19.77(4.01) 6 26.64(3.2) Ab 19.45(2.46) PE Top 0 *45.47(10.02) Aa *31.06(1.91)	.84) Ba	*31.22(9.8	*38.17 (6.76) Aa	0	Тор	XL
Bottom 0 36.41(10.56) Aa 21.78(4.9) 3 33.27(8.89) Aa 18.81(9.19) 6 30.13(14.22) Aa 14.8(2.82) S Top 0 *37.11(7.93) Aa *31.18(2.13) 3 *37.39(11.34) Aa *34.23(5.11) 6 *36.07(6.8) Aa *33.64(8.87) Bottom 0 30.15(4.3) Aa 15.82(7.88) 3 35.28(11.19) Aab 20.88(5.3) A 6 20.49(10.28) Ab 21.68(5.81) HP Top 0 *38.56(5.98) Aa *36.79(2.28) 3 *37.28(4.17) Aa *27.15(4.53) 6 *35(4.87) Aa *33.59(5.38) Bottom 0 29.92(1.96) Aa 27.38(7.26) 3 29.64(1.93) Aab 19.77(4.01) 6 26.64(3.2) Ab 19.45(2.46) PE Top 0 *45.47(10.02) Aa *31.06(1.91)).51) Ba	*29.69(10.5	*39.31(7.13) Aa	3		
3 33.27(8.89) Aa 18.81(9.19) 6 30.13(14.22) Aa 14.8(2.82) S Top 0 *37.11(7.93) Aa *31.18(2.13) 3 *37.39(11.34) Aa *34.23(5.11) 6 *36.07(6.8) Aa *33.64(8.87) Bottom 0 30.15(4.3) Aa 15.82(7.88) 3 35.28(11.19) Aab 20.88(5.3) A 6 20.49(10.28) Ab 21.68(5.81) HP Top 0 *38.56(5.98) Aa *36.79(2.28) 3 *37.28(4.17) Aa *27.15(4.53) 6 *35(4.87) Aa *33.59(5.38) Bottom 0 29.92(1.96) Aa 27.38(7.26) 3 29.64(1.93) Aab 19.77(4.01) 6 26.64(3.2) Ab 19.45(2.46) PE Top 0 *45.47(10.02) Aa *31.06(1.91)	.08) B a	*25.51(5.08	*38.81(11.62) Aa	6		
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S Top 0 *37.11(7.93) Aa *31.18(2.13) 3 *37.39(11.34) Aa *34.23(5.11) 6 *36.07(6.8) Aa *33.64(8.87) Bottom 0 30.15(4.3) Aa 15.82(7.88) 3 35.28(11.19) Aab 20.88(5.3) A 6 20.49(10.28) Ab 21.68(5.81) HP Top 0 *38.56(5.98) Aa *36.79(2.28) 3 *37.28(4.17) Aa *27.15(4.53) 6 *35(4.87) Aa *33.59(5.38) Bottom 0 29.92(1.96) Aa 27.38(7.26) 3 29.64(1.93) Aab 19.77(4.01) 6 26.64(3.2) Ab 19.45(2.46) PE Top 0 *45.47(10.02) Aa *31.06(1.91)	19) Bab	18.81(9.19	33.27(8.89) Aa	3		
3	32) Bb	14.8(2.82	30.13(14.22) Aa	6		
Bottom 6 *36.07(6.8) Aa *33.64(8.87) Bottom 0 30.15(4.3) Aa 15.82(7.88) 3 35.28(11.19) Aab 20.88(5.3) A 6 20.49(10.28) Ab 21.68(5.81) HP Top 0 *38.56(5.98) Aa *36.79(2.28) 3 *37.28(4.17) Aa *27.15(4.53) 6 *35(4.87) Aa *33.59(5.38) Bottom 0 29.92(1.96) Aa 27.38(7.26) 3 29.64(1.93) Aab 19.77(4.01) 6 26.64(3.2) Ab 19.45(2.46) PE Top 0 *45.47(10.02) Aa *31.06(1.91)	.13) Aa	*31.18(2.1	*37.11(7.93) Aa	0	Тор	S
Bottom 0 30.15(4.3) Aa 15.82(7.88) 3 35.28(11.19) Aab 20.88(5.3) A 6 20.49(10.28) Ab 21.68(5.81) HP Top 0 *38.56(5.98) Aa *36.79(2.28) 3 *37.28(4.17) Aa *27.15(4.53) 6 *35(4.87) Aa *33.59(5.38) Bottom 0 29.92(1.96) Aa 27.38(7.26) 3 29.64(1.93) Aab 19.77(4.01) 6 26.64(3.2) Ab 19.45(2.46) PE Top 0 *45.47(10.02) Aa *31.06(1.91)	.11) Aa	*34.23(5.1	*37.39(11.34) Aa	3	•	
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HP Top 0 *38.56(5.98) Aa *36.79(2.28) 3 *37.28(4.17) Aa *27.15(4.53) 6 *35(4.87) Aa *33.59(5.38) Bottom 0 29.92(1.96) Aa 27.38(7.26) 3 29.64(1.93) Aab 19.77(4.01) 6 26.64(3.2) Ab 19.45(2.46) PE Top 0 *45.47(10.02) Aa *31.06(1.91)	.3) Aab	20.88(5.3)		3		
3 *37.28(4.17) Aa *27.15(4.53) 6 *35(4.87) Aa *33.59(5.38) Bottom 0 29.92(1.96) Aa 27.38(7.26) 3 29.64(1.93) Aab 19.77(4.01) 6 26.64(3.2) Ab 19.45(2.46) PE Top 0 *45.47(10.02) Aa *31.06(1.91)	81) Ab	21.68(5.81	20.49(10.28) Ab	6		
Bottom 6 *35(4.87) Aa *33.59(5.38) Bottom 0 29.92(1.96) Aa 27.38(7.26) 3 29.64(1.93) Aab 19.77(4.01) 6 26.64(3.2) Ab 19.45(2.46) PE Top 0 *45.47(10.02) Aa *31.06(1.91)	.28) Aa	*36.79(2.2	*38.56(5.98) Aa	0	Тор	HP
Bottom 0 29.92(1.96) Aa 27.38(7.26) 3 29.64(1.93) Aab 19.77(4.01) 6 26.64(3.2) Ab 19.45(2.46) PE Top 0 *45.47(10.02) Aa *31.06(1.91)	.53) Aa	*27.15(4.5	*37.28(4.17) Aa	3	-	
3 29.64(1.93) Aab 19.77(4.01) 6 26.64(3.2) Ab 19.45(2.46) PE Top 0 *45.47(10.02) Aa *31.06(1.91)	5.38) A	*33.59(5.3	*35(4.87) Aa	6		
BE Top 6 26.64(3.2) Ab 19.45(2.46) *45.47(10.02) Aa *31.06(1.91)	26) Aa	27.38(7.26	29.92(1.96) Aa	0	Bottom	
PE Top 0 *45.47(10.02) Aa *31.06(1.91)	01) Aa	19.77(4.01	29.64(1.93) Aab	3		
	46) Aa	19.45(2.46	26.64(3.2) Ab	6		
3 *34 49(2 22) Aa *28 66(10 29	.91) Ba	*31.06(1.9	*45.47(10.02) Aa	0	Тор	PE
5 51.17(2.22) 11a 20.00(10.27).29) Ba	*28.66(10.2	*34.49(2.22) Aa	3	-	
6 *42.34(6.18) Aa *32.52(2.35)	.35) Ba	*32.52(2.3	*42.34(6.18) Aa	6		
Bottom 0 38.18(7.61) Aa 25.64(9.09)	09) Ba	25.64(9.09	38.18(7.61) Aa	0	Bottom	
3 24.97(2.4) Aab 18.63(4.39)	39) Bab	18.63(4.39	24.97(2.4) Aab	3		
6 28.42(9.72) Ab 18.4(4.63)	63) Bb	18.4(4.63	28.42(9.72) Ab	6		

There was no significant difference for photoactivation mode (p=0.8978). Averages followed by different letters (lower case in horizontal and capital letters in vertical, comparing distances within each surface and photoactivation mode) are different between them (p \leq 0.05). * Different from bottom surface in the same photoactivation mode, distance and composite resin (p \leq 0.05).

DISCUSSION

This in vitro study evaluated the effect of photoactivation mode and the distance from the LCU on microhardness Knoop (KHN) and diametrical tensile strength (DTS) of methacrylate and silorane-based composite resins. Thereunto, methacrylate-based composite resin Filtek Z250 and silorane-based composite resin Filtek P90, both from 3M ESPE were used.

In the analysis of results, it was observed that Filtek Z250 presented values of KHN equals or higher than Filtek P90. This result may be explained from the composition of each resin. Composite resin P90 presents content of 55% (in volume) of load particles in its composition, lower than the content of composite resin Filtek Z250. The increase in the content of inorganic particles results in a material with higher surface hardness.⁶⁻⁷ The

results obtained for microhardness test are according to the study performed by Lien & Vandewalle (2010),³ who also have related than the methacrylate base composite resin

presented higher compressive strength than silorane base composite resin.

Table 2. Results of Strength to Diametric Tensile (standard deviation).

Distance	Photoactivation mode	Compos	Tukey	
		Z250	P90	
0	XL	802.51(133.1)	710.99(113.51)	a
	S	639.37(175.32)	735.55(111.4)	b
	HP	669.35(175.97)	732.39(132.52)	ab
	PE	668.3(182.42)	616.61(157.54)	ab
3	XL	766.24(143.8)	654.7(257.26)	a
	S	747.34(152.4)	633.5(108.55)	b
	HP	814.8(122.1)	571.64(124.57)	ab
	PE	739.95(126.99)	643.18(113.12)	ab
6	XL	874.7(111.5)	710.23(183.86)	a
	S	653.45(146.8)	763.46(75.7)	b
	HP	759.06(140.79)	666.88(141.7)	ab
	PE	920.87(119.17)	717.48(75.08)	ab
Tukey		A	В	

There was not significant differences among the distances (p=0.0967). Averages followed by different letters (capital letters on horizontal and lower cases in vertical, comparing photoactivation mode within each distance) are different between them (p \leq 0.05).

Top surface presented higher values of KHN than bottom surface, what is according to the study performed by Aguiar et al. (2005),² who concluded that top surface presents higher hardness than bottom surface. This finding is explained because top surface is less dependent on the light intensity than bottom surface, which needs higher light intensity for better polymerization.⁸ During the photoactivation, the light emitted by the LCU passes through the body of material and suffers absorption and scattering. Consequently, the light intensity is attenuated as increases the depth, and a lower quantity of energy achieves the bottom surface.^{9,10}

The distance from the LCU (0mm, 3mm and 6mm) did not have statistically significant difference in the top surface microhardness. However, for the bottom surface was observed a significant statistical difference, in which the lower values of KHN when photoactivated at 6 mm. This finding may be explained through the existence of a relation inversely proportional to the light intensity and the square of the distance between the light source and the surface irradiated. Through this phenomenon is explained because the increase of distance may provoke lower hardness of material.¹¹ This result is according to the study carried out by Aguiar et al. (2005),² who reported that in

the distance of 8mm there was also a significant decrease of composite resin hardness. Through this result, it was observed that the distance from the light curing source influences considerably the effectiveness of polymerization, and it is an important factor, mainly in very deep restorations, and in these cases, Atmadja et al. (1990)¹² and Prati et al. (1999)¹³ have recommended in their studies the increase of photoactivation time for a better polymerization.

Diametrical tensile strength is a test performed in vitro and considered a good indicator of behavior for restorer material because it simulates the loads which focuses in this material during the mastication.⁶ In the present study, it was observed that higher values were obtained for Filtek Z250, while Filtek P90 presented the lower values. This behavior may have its explanation in the way that the inorganic and organic phase and the bond agent react in each one of evaluated systems. Silane is a bi-functional molecule employed as bond agent because it forms chemical bonds between load particles and organic matrix, improving the distribution of tensile which occurs between these phases during the masticatory effort, and thus given higher resistance.13 For methacrylate composite resins, the evidences suggests that the use of silane results in a composite material with better mechanical properties because the inorganic and organic phases.

However, in silorane base resins, the silane layer interact with silorane matrix in a different way and results in a less resistant bond, and does not present the same reinforce obtained by methacrylate base resins.³ Other possible explanation for lower values of strength to DST obtained by composite resin Filtek P90 may be the fact of silorane exhibiting a slower polymerization reaction, and by this reason it needs higher quantity of energy to start the polymerization of material. ^{14,15} In case in which the slower polymerization occurs, the configuration of polymeric chain formed would be more linear, what may justify a lower cohesive force of this material.

CONCLUSION

From the analysis of data and the discussion in this study, it is possible conclude that:

- 1. Photoactivation modes do not have influence in microhardness and diametrical tensile strength of materials evaluated.
- 2. Methacrylate base composite presented better mechanical properties than silorane base composite.
- 3. 6 mm from de light-curing unit resulted in significant reduction of bottom surface hardness for both materials.

ACKNOWLEDGEMENTS

This study was sponsored by the Programa Institucional de Bolsas de Iniciação

Científica (PIBIC) through the grant of scholarship. The authors are indebted to the Department of Dental Materials, School of Dentistry of Piracicaba – Brazil, for allowing the use of their Universal Testing Machine.

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