

INFLUENCE OF POLISHING PROTOCOL ON FLEXURAL PROPERTIES OF SEVERAL DENTAL COMPOSITE RESINS

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ABSTRACT

The aim of this study was to determine the influence of the finishing protocol (FP) on flexural properties of several composites (CR). Twenty composite samples (25x2x2 mm) were prepared: G1 Helimolar[®]; G2 Filtek[™] Z350; G3 Tetric[®] N Ceram, G4 Point 4[™], G5 Premisa[™]; G6 Esthet.X[®] HD, G7 ice, G8 Vit-L-escence[®], G9 Grandio[®], G10 TPH[®]3, G11 Amelogen[®] Plus, G12 Brilliant Enamel; G13 Filtek[™] Z100 and randomly divided into four groups according to the finishing system: C control, J Jiffy[®], SS Super Snap[®], AA Astropol[®] /Astrobrush[®]. Each sample was polished for 10 seconds with each sequence instrument, and stored in distilled water for 24 hours, after which

a three-point flexure test was applied to determine flexural strength (FS) and modulus (Flexural modulus). Data were analyzed using a two-way multivariate ANOVA and means were compared with Tukey's test. Results were: FS level CR $p=0.000$ with significant differences. FS level FP $p=0.093$ with significant differences. In order: FM level CR $p=0.00$ with significant differences. FM level PS $p=0.001$; with significant differences. Under the study conditions, the polishing systems based on silicone rubber decreased the flexural properties of composite resins.

Key words: composite resins; elastic modulus; dental polishing.

INFLUENCIA DEL PROTOCOLO DE PULIDO SOBRE LAS PROPIEDADES FLEXURALES DE VARIAS RESINAS REFORZADAS

RESUMEN

El objetivo de este trabajo fue determinar la influencia del protocolo de pulido sobre las propiedades flexurales de varios composites. Se prepararon veinte probetas de composite (CR) (25x2x2 mm) en cada grupo: G1 Helimolar[®]; G2 Filtek[™] Z350; G3 Tetric[®] N Ceram, G4 Point 4[™], G5 Premisa[™]; G6 Esthet.X[®] HD, G7 ice, G8 Vit-L-escence[®], G9 Grandio[®], G10 TPH[®]3, G11 Amelogen[®] Plus, G12 Brilliant Enamel; G13 Filtek[™] Z100 y se dividieron aleatoriamente en cuatro grupos según el sistema de pulido (SP) utilizado: C control, J Jiffy[®], SS Super Snap[®], AA Astropol[®] /Astrobrush[®]. Cada probeta se pulió durante 10 segundos con cada secuencia de instrumentos, se almacenó en agua destilada durante 24 hs y se aplicó un ensayo de resistencia flexural de tres puntos a fin de determinar la

resistencia (RF) y el módulo (MF) flexural. Los valores obtenidos fueron analizados con un ANOVA multivariado de dos vías y comparación de medias de Tukey. Los resultados obtenidos fueron: nivel de RF para CR $p=0,000$ con diferencias significativas. RF nivel SP $p=0,093$ con diferencias significativas. Para MF a nivel de CR $p=0,000$ con diferencias significativas. MF a nivel SP $p=0,000$, con diferencias significativas. En las condiciones de este trabajo se puede concluir que el uso de sistemas a base de gomas siliconadas disminuyen las propiedades flexurales de las resinas reforzadas restauradoras. : composite resins; elastic modulus; dental polishing.

Palabras clave: resinas reforzadas; módulo elástico; pulido dental.

INTRODUCTION

Composite resin is universally accepted as an anterior direct restorative material, even for large restorations. In posterior restorations its application is becoming more popular, probably because of its high esthetic potential, acceptable longevity, tissue preservation potential, tooth structure bonding, low

temperature conductivity and increased demand from patients¹. Initially these materials had many disadvantages, such as high polymerization shrinkage; occasional patient-referred post-operative sensitivity and accelerated wear in restorations, mainly located in the molar area, leading to loss of occlusal and proximal anatomy. A meticulous

restoration technique, the right case selection, improvement in material formulations and better knowledge of their performance have enabled safe use of these materials.

When considering desirable properties for clinical performance, mechanical properties are described as very important, especially because they are closely related to the long-term success of these restorations. Improvement in mechanical properties in recent formulations has led to increased toughness and resistance to abrasion and attrition wear². Mechanical properties depend mainly on composite microstructure and composition; therefore filler amount, size, morphology and distribution³ are critical for composite selection. In addition, variations in matrix chemistry should be taken into account, since it has been reported that they could significantly affect flexural strength and modulus. Flexural modulus means material stiffness, which is important because it influences composite selection in high stress situations, such as medium-large sized restorations in proximal or occlusal locations, or for incisal angle replacement or when used to replace cusps⁴.

In a three-year clinical study², 102 class 4 restorations made with 4 different composite formulations were evaluated and a correlation between mechanical properties and clinical performance was found, which is an association between wear, modulus and defect size.

Clinical procedures that may damage or prematurely harm restorations should be accurately performed because the primary cause of failure during the first five years is related to technique and material selection⁵. Optimal finishing and polishing of composites is one of the most important steps when performing a restoration, since it not only results in optimal esthetics, but also favors gingival health, restoration of marginal integrity over time and patient comfort^{6,7}, as well as increasing resistance to pigmentation and wear^{8,9} and possibly influencing restoration longevity. Surface roughness creates a favorable microenvironment for bacterial adhesion and growth, which enables the development of secondary caries, gingival inflammation and restoration staining. Polishing aims to reduce surface roughness and lines progressively until they are smaller than visible light wavelength. A traumatic technique might overheat and damage a composite surface,¹⁰⁻¹² resulting in accelerated

wear. This research group has published previous data evaluating how filler morphology, matrix composition and finishing protocol correlate with flexural properties and mass loss in several composites¹³. We concluded then that all variables evaluated correlate with a stronger weight on modulus.

There is no clear evidence of how different clinical polishing protocols might affect the flexural properties of composite resins reinforced with different fillers; thus, the aim of this study was to evaluate the effect of three polishing protocols on flexural properties of thirteen restorative composites.

MATERIALS AND METHODS

Thirteen groups of twenty samples of light cured composite resins, shade A2, from eight manufacturers, were prepared: G1 Heliomolar (Ivoclar-Vivadent, Schaan, Liechtenstein), G2 Filtek Z350 (3M/ESPE. St. Paul, USA), G3 Tetric N Ceram (Ivoclar-Vivadent, Schaan, Liechtenstein), G4 Point 4 (KERR®- Sybron dental Specialties, Orange, USA), G5 Premisa (KERR- Sybron dental Specialties, Orange, USA), G6 Esthet.X HD (DENTSPLY-Caulk, Milford, USA), G7 Ice (SDI Limited. Victoria, AU), G8 Vit-L-escence (Ultradent Products, INC. South Jordan, USA), G9 Grandio (VOCO America INC. Sunnyside, USA), G10 TPH3 (DENTSPLY-Caulk, Milford, USA), G11 Amelogen Plus (Ultradent Products, INC. South Jordan, USA), G12 Brilliant Enamel (Coltène Whaledent, Altstätten, Switzerland), G13 Filtek Z100 (3M/ESPE. St. Paul, USA). They were randomly divided into four groups of five samples each according to the polishing protocol to be applied: C control, J Jiffy (Ultradent Products, INC. South Jordan, USA), SS Super Snap (Shofu Dental Corporation, San Marcos, USA), AA Astropol /Astrobrush (Ivoclar-Vivadent, Schaan, Liechtenstein-batch H32042). Samples were prepared according to ISO Standard 4049 for flexural strength and ANSI/ADA standard 27, using a standardized 25x2x2 mm aluminum mold and verified by means of a digital micrometer 500 (Mitutoyo Corporation, Japan) with an accuracy of 0.01 mm. Composite was placed in 2 mm increments, each of which was light cured for 40 seconds using an Astralis 3 (Ivoclar-Vivadent, Schaan, Liechtenstein) unit at 600 mW/cm². Samples were normalized using moist 400 grit sandpaper. One hour later each sample was submitted to the

Table 1: Flexural strength and modulus.

	Flexural strength in MPa	Flexural modulus in GPa
Equations	$\sigma=3Fl/2bh^2$	$E=l^3F/4bh^3d$
F maximum load (Newton). l distance between supports, millimeters. b width at the centre of the specimen, millimeters. h height at the centre of the specimen, millimeters. d deflection due to load, millimeters.		

Table 2: Classification of the composite according to filler morphology.

Group and Composite	Filler Morphology
G1 Heliomolar	Pre-polymers
G2 Filtek Z350	Spherical-conglomerates
G3 Tetric N Ceram	Irregular + pre-polymers
G4 Point 4	
G5 Premisa	
G6 Esthet.X HD	Irregular
G7 Ice	
G8 Vit-L-escence	
G9 Grandio	
G10 TPH3	
G11 Amelogen Plus	Irregular + pre-polymers
G12 Brilliant Enamel	
G13 Filtek Z100	Spherical

Table 3: Descriptive statistics and multiple comparisons for flexural strength at composite level.

Composite	Mean	Typical deviation	T-HSD
G1 Heliomolar®	87.76	9.45	C
G2 Filtek™ Z350	96.77	21.48	BC
G3 Tetric® N Ceram	106.53	9.56	B
G4 Point 4™	104.16	18.40	B
G5 Premisa™	96.61	9.55	BC
G6 Esthet.X® HD	120.03	11.66	A
G7 ice	120.51	18.56	A
G8 Vit-L-escence®	119.38	14.39	A
G9 Grandio®	124.70	14.87	A
G10 TPH®3	120.80	10.37	A
G11 Amelogen® Plus	115.12	16.85	AB
G12 Brilliant Enamel	119.74	19.08	A
G13 Filtek™ Z100	135.21	18.56	A
Mean (MPa). T-HSD: Tukey's test. Equal letters indicate homogeneous groups			

polishing protocol, applying 10 seconds per step and following the manufacturer's instructions with a handpiece at 10,000 rpm NSK (NAKANISHI, Kanuma, Japan), being very careful to maintain water refrigeration and not to press too hard. Samples were stored in distilled water at 37°C for 24 hours. Sample dimensions were determined, after which they were submitted to a three point flexural test (ISO4049/2000 - 27 ANSI/ADA specification) using a universal testing machine 1011 (INSTRON®, Norwood, USA) with a crosshead speed of 1 mm/min until fracture.

Data were recorded and processed applying the equations in Table 1 in order to determine flexural strength and modulus for each sample. Statistical analysis was performed using two-way ANOVA, and DHS Tukey test was applied for multiple comparisons. (Statistical Package for the Social Sciences 15.0®).

Two series of samples of each composite resin were prepared and metalized with gold and argon laser for SEM observation at the Advanced Microscopy Center, University of Buenos Aires, Argentina, with a Supra 40 Scanning Electron Microscope, Carl Zeiss, Germany, with a field emission gun. Micrographs were taken and composites were classified into five groups: spherical, spherical-conglomerates, irregular, irregular + pre polymers and pre-polymers. (Table 2).

RESULTS

Flexural strength: descriptive statistics (mean and standard deviation) are shown in Table 3. ANOVA for flexural strength (FS) showed statistical differences among groups $p < 0.001$ (Table 4). HSD Tukey (Table 3) proved composites reinforced with spherical (G13 Filtek™ Z100) and irregular particles (G9, G7, G10, G6, G12, G8 and G11) to have higher flexural strength than composites with mixes of pre-polymerized/ irregular (G3, G4 and G5), spherical conglomerates (G2 Filtek™ Z350) and pre-polymerized particles (G1 Heliomolar®). When flexural strength was analyzed related to polishing system, ANOVA showed significant differences among groups $p < 0.05$ (Table 4). Descriptive statistics are shown in Table 5 with mean and typical deviation expressed in MPa. HSD Tukey test showed that there was no significant difference among J, SS and AA and only AA differed significantly from C. Composite-polishing

system interaction on flexural strength showed $p=0.416$, indicating that flexural properties are independent from it.

Modulus: ANOVA performed for flexural modulus showed significant differences among materials $p<0.001$. Descriptive statistics (mean and standard deviation in GPa) (Table 5) were $G13:15.03(1.09)=G9:14.50(1.50)>G11:9.79(0.52)=G10:9.76(1.27)=$

$G2:9.72(2.20)\geq G12:8.89(0.4)=G7:8.56(0.73)\geq G4:7.98(1.01)=G6:7.88(0.46)=G8:7.57(0.50)=G3:7.19(0.66)\geq G5:6.53(0.71)>G1:4.65(0.38)$. When modulus was analyzed with relation to polishing system, ANOVA showed significant differences among groups, $p<0.001$ (Table 6). Descriptive statistics are shown in Table 7 with mean and standard deviation expressed in GPa.

Table 4: Tests of Between-Subjects Effects (ANOVA).

Source	Dependent Variable	Sum of Squares	Df	Mean Square	F	Sig.
Composite	FS	43520.192	12	3626.683	15.736	<0.001
	M	2020.912	12	168.409	273.815	<0.001
Polishing	FS	1964.956	3	654.985	2.842	0.039
	M	21.394	3	7.131	11.595	<0.001
Composite* Polishing	FS	8622.865	36	239.524	1.039	0.416
	M	105.083	36	2.919	4.746	<0.001
Error	FS	47937.216	208	230.467		
	M	127.930	208	0.615		
Total	FS	3414321.239	260			
	M	23721.978	260			
Corrected Total	FS	102045.229	259			
	M	2275.319	259			

FS: flexural strength. M: modulus df: degrees of freedom

Table 5: Descriptive statistics and multiple comparisons for flexural modulus at composite level.

Composite	Mean (GPa)	Typical deviation	T-HSD. $p=.05$
G1 Heliomolar®	4.65	0.38	F
G2 Filtek™ Z350	9.72	2.20	BC
G3 Tetric® N Ceram	7.19	0.66	DE
G4 Point 4™	7.98	1.01	D
G5 Premisa™	6.53	0.71	E
G6 Esthet.X® HD	7.88	0.46	D
G7 ice	8.56	0.73	CD
G8 Vit-L-escence®	7.57	0.50	D
G9 Grandio®	14.50	1.50	A
G10 TPH®3	9.76	1.27	B
G11 Amelogen® Plus	9.79	0.52	B
G12 Brilliant Enamel	8.89	0.40	C
G13 Filtek™ Z100	15.03	1.09	A

T-HSD: Tukey's test. Equal letters indicate homogeneous groups

Table 6: Descriptive statistics and multiple comparisons for flexural strength at polishing level.

Group	Composite	Mean (GPa)	Typical deviation	T-HSD. $p=.05$
C	Control	117.54	20.33	A
J	Jiffy®	111.53	21.20	AB
SS	Super Snap®	111.92	20.57	AB
AA	Astropol/Astrobrush	110.48	16.66	B

T-HSD: Tukey's test. Equal letters indicate homogeneous groups

Table 7: Descriptive statistics and multiple comparisons for flexural modulus at polishing level.

Group	Composite	Mean (GPa)	Typical deviation	T-HSD. $p=.05$
J	Jiffy®	8.65	2.50	C
SS	Super Snap®	9.16	3.10	AB
AA	Astropol/Astrobrush	9.06	3.25	B
C	Control	9.45	2.95	A

T-HSD: Tukey's test. Equal letters indicate homogeneous groups

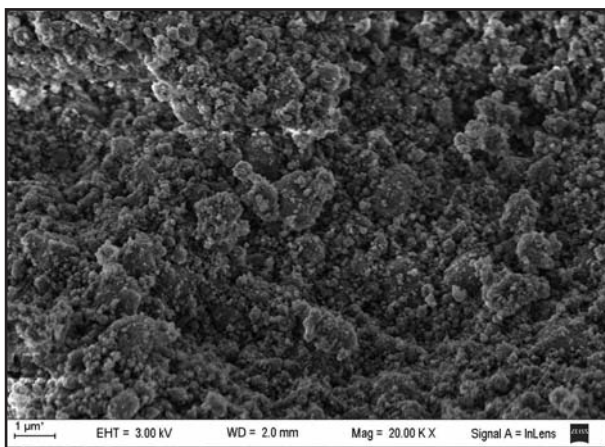


Fig. 1: SEM image at 10,000x of Filtek Z100 composite resin reinforced with spherical fillers.

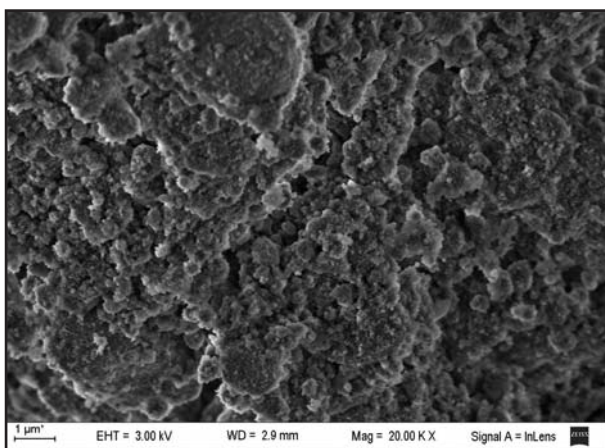


Fig. 2: SEM image at 10,000x of Filtek Z350 composite resin reinforced with spherical conglomerate fillers.

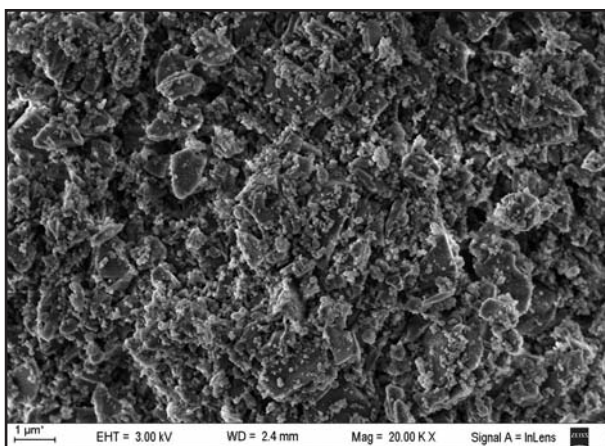


Fig. 3: SEM image at 10,000x of composite resin reinforced with irregular fillers. From right to left upper and lower images of Esthet.X® HD, Ice, Vit-L-escence, Grandio, TPH3 and Amelogen Plus.

Groups were as follows: C= SS \geq AA: > J. HSD Tukey test proved that all experimental groups showed lower modulus values than the control, although SS had no significant difference. Jiffy® had the lowest values and differed significantly. Composite-polishing system interaction on flexural modulus showed $p < 0.001$ (Table 4), indicating that flexural modulus is dependent on it.

Figures 1-5 show SEM photographs taken of each type of composite and their classification in terms of filler morphology.

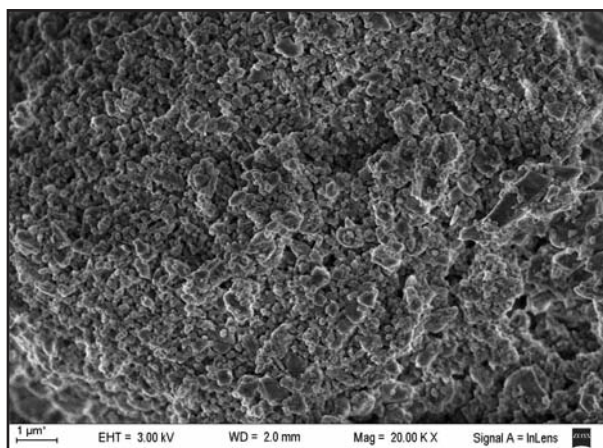


Fig. 4: SEM image at 10,000x of composite resin reinforced with irregular fillers + pre-polymer fillings. From right to left upper and lower images of Point 4, Premisa, Tetric N Ceram and Brilliant Enamel.

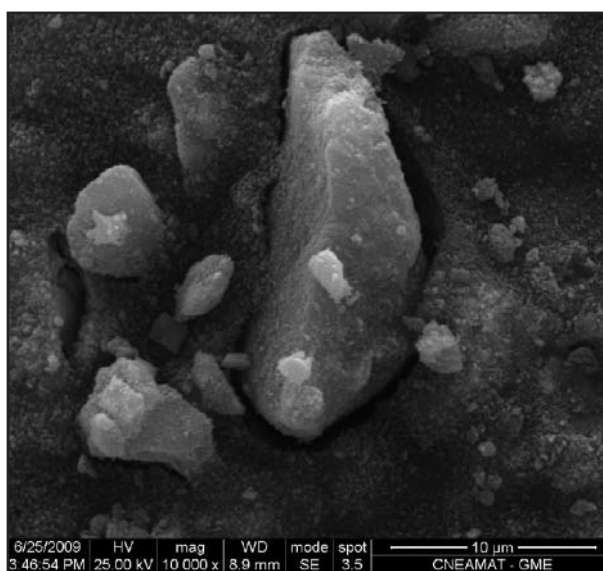


Fig. 5: SEM image at 10,000x of Heliomolar composite resin reinforced with pre-polymers.

DISCUSSION

Two out of the three polishing protocols evaluated in this investigation are based on silicone impregnated with varied grain size abrasives, mostly silicon carbide (Jiffy and Astropol). The third (Super Snap) is based on flexible paper discs impregnated with silicon carbide and aluminum oxide.

In order to calculate flexural performance, we carried out a three point stress test which applies one force in one sense that generates three types of loads (shear, tensile and compressive)¹⁴. Some authors consider that these results cannot be transferred to clinical situations, primarily because failure distribution is different. However, the model we applied in order to determine mechanical properties here is accepted worldwide because it is easy to reproduce, serves in terms of comparative purposes and represents material behavior in biomechanical efforts and determines critical performance during function^{15,16}. In terms of FS, all evaluated composites comply with ISO Standard 4049. However, there were some differences among commercial brands. In our study, composites with spherical filling proved to be similar to composites with irregular particles. Small variations might be given by filler quantity and mean size because the manufacturing processes are essentially the same. Therefore, it can be speculated that recent formulations with optimized matrix containing nanometric spherical filler do not seem to improve FS. It could be considered that variations (although not significant) in FS occur because irregular particles concentrate stress at dead angles in the polymerized matrix. On the other hand, differences in monomer proportions might significantly change mechanical properties⁴, where a slight TEGDMA increase might cause a strength reduction and a UEDMA increase would turn into an increase. Furthermore, BisGMA variations do not seem to affect the final result. It can thus be speculated that TEGMA content may be responsible for preventing Grandio high filling content and spherical particles in Filtek Z-100 from being significantly superior in mechanical properties to the other irregular particles composites. Finally, the lower part of the table shows the results for pre-polymerized + irregular reinforced composites (Tetric N Ceram, Point 4, Premisa), spherical conglomerate (Filtek Z-350) and pre-polymerized (Heliomolar). It could be explained by several factors such as their lower sized fillers, which

increase the surface to be covered by the matrix, leading to less filler content. Also more silane could be present and diminish the polymerization degree, modifying their mechanical properties¹⁸. All composites, with the exception of Tetric N Ceram, have TEGMA in their composition and smaller particles are less effective in preventing crack propagation because they are easier to go around. Regarding composites with pre-polymerized particles, it could be speculated that the high conversion degree might affect bond strength with unpolymerized matrix, leading to lower mechanical properties. In Filtek Z-350, it can be inferred that although the filler keeps its spherical shape, sintered nanometric particles are not bonded enough to alter the course of the fracture, causing a conglomerate separation. Our group has previously reported a part of this research focused on filling morphology, matrix and polishing protocol correlation with flexural properties and surface loss¹³. We found that flexural strength, flexural modulus and surface loss were significantly correlated to the composition and morphology of these composite resins. The highest influence was found for modulus at 85.7%, while it was lower for resistance and surface loss, at 41% and 36.3% respectively. Although that paper also analyzed the influence of the matrix composition, we reported, for example, that spherical conglomerates negatively influence mechanical properties. On the other hand, independent spherical particles might turn into a more regular structure that could help distribute tension, thus increasing properties. This might suggest that properties are influenced not only by filler morphology, but also by filler distribution. Finishing refers to the restoration's adaptation to the tooth and aims to obtain good contour, occlusion, healthy relationship with adjacent teeth and a smooth surface⁹, while polishing refers to the elimination of irregularities in order to create a surface which is as smooth as possible, to minimize gingival irritation, biofilm accumulation, pigmentation, and recurrent formation of caries¹⁸⁻²¹. We carried out the whole process using water cooling in order to avoid some factors that may affect resins when the procedure is performed without water cooling²². It was also finished a few minutes after initial curing, since lack of polymerization and surface roughness in newly cured composite resins²³ have been rejected in recent formulations^{24,25}.

There are more variables that might influence restoration properties, such as operator, time, humidity and matrixes were controlled, pressure applied to polish the samples. That pressure might increase tension in the sample, and even transmit pneumatic handpiece vibrations, which might induce some kind of initial defect in the material. All these variables were controlled by limiting all procedures to a single operator.

The efficacy of abrasive systems is related to the flexibility of the supporting material. Abrasive hardness in this case is very close in the different systems (2500 Knoop H Kg/mm² silicon carbide and 2100 Knoop H Kg/mm² aluminum oxide), instrument geometry and application. Differences between flexible discs and silicone rubber reside in the fact that discs wear filler particle and matrix in a similar way⁷, whereas rubbers wear matrix by plastic deformation, leading to temperature increase and either leaving particles on the surface or removing them, both of which leave irregularities that translate

into a less bright surface. They also create defects where the cracks initiate, leading the material to its strength limit. Moreover, physical stress caused by temperature increase might result in the formation of micro-cracks, micro-pores or interface spaces between matrix and filler, which might affect physical properties⁹. Our results differ from those reported by Gordan et al. 2003¹⁰, who found an increase in FS when polishing maneuvers were carried out.

CONCLUSIONS

Under the conditions of this study, flexural properties of composite resins were affected by the polishing systems tested. FS is higher in composites reinforced with spherical and irregular particles. Flexural strength is diminished when the Astropol/ Astrobrush system is used. Regarding stiffness, Filtek Z100 and Grandio proved to be superior to the other resins tested. However, both resins showed lower stiffness when systems based on silicone rubber such as Astropol/Astrobrush and Jiffy were used.

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