Ceromers and resins: Comparing mechanical properties.

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Abstract. *10 samples of Cerometer Adoro-Ivoclar, Gradia-G.C packable resine P60-3M (fotopolimerized and heat treated) and SureFil-Dentsply (fotopolimerized and heat treated) were made to conduct compression and Vickers hardness tests for a total of 120 samples. Values measured and compared were ultimate stress, Young`s modulus, and Vickers Hardness Number (VHN). Results pointed up that heat treated P60 showed the highest ultimate stress (273 MPa), and Adoro the lowest (123,7 MPa), P60 showed the highest hardness (93 VHN) and Gradia the lowest (35 VHN). Related to Young`s modulus, Surefil has the highest (535,1 kPa) and Gradia the lowest (128,5 kPa). This in vitro mechanical test offers the possibility to know and compare the inner properties of several comerical resins against ceromer material.*

Keywords*: Mechanical characterization, ceromer composite resin, Vickers Hardness, ultimate stregth, packable resines*

1. INTRODUCTION

The analysis of mechanical properties provides with valuable information. We agree that extrapolations of these in-vitro findings to the clinic practice are impossible due to the complex dynamic and environment of the oral cavity. However knowing the inner properties for each material is important. In this sense we believe that providing basic information of these materials could help understand future in-vivo studies.

Mechanical properties of restorative dental materials (resins and ceromers among others) has advanced enourmously in the previous years due to estetic and functional necessities to restore posterior tooth optimizing changes in properties of dental materials including their curing processes (Torres, 2001). Amalgam is currently the most widely used dental filling material worldwide for the restoration of posterior teeth because of its straightforward handling procedures, well-tested material properties, and clinical success, which has been documented for over a century. According to Bog L., et al (2007) composite resines can be classified according to density in the following order:

- Low density (Fluid)
- Medium density (Conventionals)
- High density (Packable)
- Super high density (Ceromers)

Material cost and simple application techniques also make it the most economic dental filling material (Logercio, 2006). The demand for tooth-colored restorations, for esthetic purposes, has grown considerably during the last decade (Sadowsky, 2006). Metal fillings, directly or inderectly, were the only restoration option for many years. However, esthetic considerations made dental materials develop (Torres, 2001). With this in mind, photocured resins are considered an alternative to metal fillings to restore posterior tooth. According to Sheibenbogen et al (1999), 90% of dental restaurations were clinically acceptable after a two years evaluation. In 1991 Barnes et al reported 90% success after five years and 77% after 8 years. According to Sheibenbogen et al, (1999) the use of metal fillings for posterior tooth, using direct technique, is restricted due to insufficient mechanical properties; failure is caused by deformation and low wear resistance causing loss of anatomical form under abrasion in the masticating process. According to this, an indirect technique using composites was developed, which is able to withstand compressive, tensional, and torsional loads present in the masticatory cicle, giving better occlusal and functional resistance wear, adaptation at the gingival level, and providing a smoother surface. However, results with resines using indirect method were not what the dental community hoped for and ceromers were developed when compared to resines, due to a higher degree convertion obtained with polimerization processes such as photoactivation, heat and /or Nitrogen atmosphere (Cesar et al., 2001). Given this materials evolution, ignorance and lack of scientific support for mechanical composite resins properties subjected to additional heat treatment, i.e. in boiling water after being photopolymerized to be used with indirect techniques to restore poterior teeth. The aim of this work was to compare the mechanical properties of two ceromers (Gradia-GC and Adoro-Ivoclar) with two packable resines photopolimerized differently with heat treatment (SureFil-Dentsply, P60-3M).

2. Materials and Methods

Even though analysis of mechanical properties provides extermely valuable information and in-vitro testing can simulate the materials to the comprehensive and abrasive conditions of the oral cavity, the clinical qualification can only be obtained in clinical studies (Cunningham, 1989).

10 samples of Cerometer Adoro-Ivoclar, Gradia-G.C packable resine P60-3M (fotopolimerized and heat treated) and SureFil-Dentsply (fotopolimerized and heat treated) were made to conduct compression and Vickers hardness tests for a total of 120 samples. Values measured and compared were ultimate stress, Young`s modulus, and Vickers Hardness Number (VHN).

10 samples were made for compression test and 10 samples for hardness test in each group. Specimens had dimmensions of 8 mm in height and 4 mm in diameter for compression and 2 mm in height and 7 mm in diameter for hardness test according to Marghalani H. et al (2004) and Verane Y. et al (2005).

The independent variables were ultimate strenght, Young`s modulus, and Vickers hardness. Specimens that did not polimerized completely and / or presented visible rugosity were eliminated. To make the specimens, Teflon molds were fabricated for each test.

Every mold was smeared with a petrolate thin film and placed vertically on a microscope´s stage. Samples were fabricated with two (2) mm layers according to each manufacturer´s recommendations and material was packed into the upper opening in bulk with a condenser in order to minimize the inclusion of air bubbles into the specimen. A Mylar stripe was placed on top of each finished sample and slight pressure was applied to create a smooth surface.

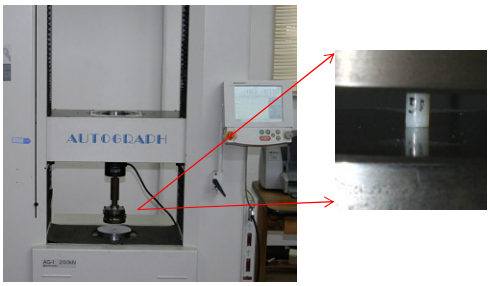
After initial photo polimerization for ten seconds (*GC Steplight SL lamp* for Gradia and *Quick lamp* for Adoro), Cerometer samples (Gradia and Adoro) were submitted to a heat treatment procedure in their respective furnace (*Labolight LV III* for Gradia for about five minutes and *Lumamat 100* for Adoro for about 25 minutes). Packable resine samples (3M´s P60 and Dentsply´s SureFil) were photpolimerized with a LED TPC55 lamp for twenty seconds. Half of those samples were heat treated in boiling water for seven minutes and stored in demineralized water at room temperature for eight days following procedures reported by several authors (Porto et al, 2004; Mandikos et al., 2001; Veranes et al., 2005).

Packable resines (P60 and Surefil) were divided in two groups. The first was polimerized in the convetional form (*LED lamp* for 20 seconds every time). The second group had an adittional heat treatment, which consisted in boiling water the samples for seven minutes. Ceromers were processed according to manufacturer´s recommendations and appropiate equipment.

Samples elaboration in this study was done according to ISO 4049 (Polymer-based filling, restorative and luting materials Standard). All coupons were observed and analized for quailified external personnel to the study.

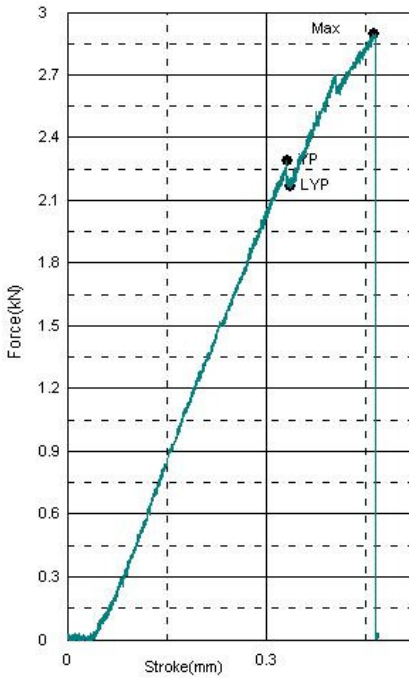
Compresion tests were made in a Shidmazu Autograph AG-I 250 kN universal testing machine with a 10kN load cell at a 0.05 mm/min speed. The machine had been calibrated just one month before by the supplier. For data adquisition, Trapezium 2.23 was used taking measurements every 50 milliseconds. Figure 1 shows compression setup.

Figure 1B. Compresion test setup



It is important to mention that some samples showed what we called “fake recovery”. This occurs when a sample still withstands the load after partially loosing its original geometry. For this particular application, even though a chipped filling may still work from a structural point of view, esthetic and most likely pain considerations render the filling as a must-replace-it. This was taking into account when selecting the ultimate compressive stress and points to draw a proportional line and calculate Young`s modulus. Figure 2 shows an example of that case where the ultimate compressive stress is the one about 2,3 kN and not the one at 2,9 kN.

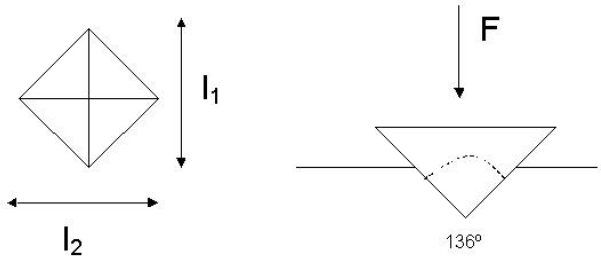
Figure 2. Sample showing fake recovery.



Hardness tests were conducted in a Shimadzu N° 4989 hardness machine using a Vickers diamond indentor with 500gf load for 15 seconds following procedures reported by several authors (Lovell et al., 2001; Davidson et al., 1997; Martins et al. 2008). Three measurements were taken for every sample (Martins et al. 2008) and a VHN was calculated according to equation one (Eq. 1):

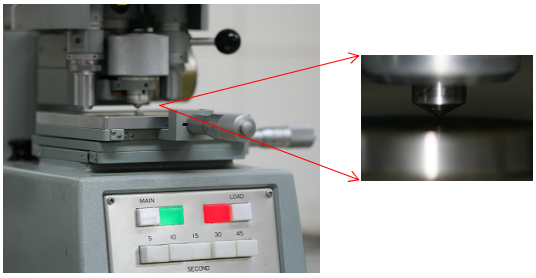
Figure 3. Schematics for VHN test

$$VHN = \left(\frac{1854,4F}{\left(\frac{l_1 + l_2}{2} \right)^2} \right) \quad \text{(Eq. 1)}$$



Where F is the load in grams and l_1 and l_2 are the lengths of the diagonals left by the indentor as shown in Figure 3. Figure 4 shows the Vickers hardness setup.

Figure 4. Vickers hardness setup.



It is important to say that both types of tests were conducted at room temperature controlled by commercial AC units.

For statistical process and data treatment, results were averaged and their standard deviation was calculated.

In a legal framework, this work complies with Colombian law 8430 from 1993 requirements which regulates health related tests and research.

2.1. Results

One variable analisys values are presented with mean and standard deviation.

Table 1 presents ultimate compressive strenght mean and standard deviation values for the before mentioned materials.

Table 1. Ultimate compressive strenght mean and standard deviation comparisson for materials tested at 23C. (*HT: Heat treatment*)

SAMPLE #	MATERIAL	MEAN (MPa)	STANDARD DEVIATION
1-10	Adoro	123,7	38,4
11-20	P60 with HT	273,26	35,79
21-30	P60 w/o HT	254,71	51,63
31-40	Gradia	172,87	35,60
41-50	SureFil with HT	268,96	21,02
51-60	SureFil w/o HT	242,75	43,30

The material that presented the highest ultimate compressive strenght mean was P60 with HT at 273,26 MPa closely followed by SureFil with HT with 267 MPa. The lowest values were given by ceromer Adoro at 123,7 MPa and Gradia at 172,87 MPa.

Table 2 presents Young`s Modulus mean and standard deviation values for the afore mentioned materials, whereas Table 3 presents VHN mean and standard deviation values. It was calculated using values between two choseen points, always picking the best linear fit.

Table 2. Young`s Modulus mean and standard deviation comparisson for materials tested at 23C.

SAMPLE #	MATERIAL	MEAN (kPa)	STANDARD DEVIATION
1-10	Adoro	261,1	20,3
11-20	P60 with HT	434,7	15,8
21-30	P60 w/o HT	504,6	26,5
31-40	Gradia	128,5	10,1
41-50	SureFil with HT	535,1	15,2
51-60	SureFil w/o HT	397,7	24,2

Table 3 VHN mean and standard deviation values.

SAMPLE #	MATERIAL	MEDIA (VHN)	DESVIACION ESTANDAR
1-10	Adoro	51,00	2,86
11-20	P60 with HT	93,62	2,83
21-30	P60 w/o HT	93,13	2,11
31-40	Gradia	35,35	2,08
41-50	SureFil with HT	85,97	5,37
51-60	SureFil w/o HT	69,78	2,77

ANOVA was made to evaluate the hypohtesis, “Ceromers are harder and have higher ultimate strengh fracture than packable resines” using material type (ceromer and resine) as independent variables. For ultimate strengh fracture by material type, significant differences were found as shown in Tables 4 and 5.

Table 4. ANOVA for ultimate strengh fracture by material type

Variation Source	ANOVA				
	SS	FD	SM	F	Prob>F
Between groups	117629.067	5	23525.8133	13.21	0.0000
Within groups	96199.7633	54	1781.4771		

Table 4. ANOVA for VHN by material type

Variation Source	ANOVA				
	SS	FD	SM	F	Prob>F
Between groups	28970.4281	5	5794.08561	563.91	0.0000
Within groups	554.840089	54	10.2748165		

SS: Squares Sum; FD: freedom degrees, SM: Square Mean; F: test F

ANOVA test for VHN, depending on the material type, showed statistically significant differences. ($p = 0.0000$)

2.2. Discussion

Packable resins composites were born due to the progressive restorative materials development to fill teeth cavities after a cleaning preparation. Resines have different characteristics, mainly in size and distribution of particles, which causes marked differences in mechanical and physical properties (Loguercio A.D., 2006). Ceromers are heat optimized polymers with ceramic particles. That is why they are known as seventh generation composites. They are used for restoration using indirect technique.

In an effort to address concerns surrounding the propertied of these composite esthetic materials for posterior restorations, efforts have been directed toward increasing the filler content in the composite matrix and reducing the filler particle size (Knobloch, 2002). The chemistry, method of polymerization, and bond between the filler and matrix has also been studied (Ruyter, 1982; Ferracane, 1985). More recently, packable composites have been introduced with larger filler particles than microfill and hybrid composites. In addition, the use of irregular filler particles of different sizes, irregularly shaped glass fibers, and rough porous fillers have been incorporated in an attempt to achieve packability handling of the composite material (Knobloch, 2002). Ceromers, light-, heat-, or vacuum-polymerized laboratory processed particulate composite materials, use new polymer formulations with improved filler particle distribution. Therefore, they have been promoted as a hybridization of composite and ceramic technologies, although they are essentially still a composite resin matrix with differing filler components (Kurker, 2006).

Packable resines and ceromeres are materials that are known for presenting an inorganic matrix usually composed by Zirconium, Silica, Barium, Flour, among others, with particle size ranging from 0.5 y 30 μm. Furthermore they use copolimers which are micro stuffing shattered and ground composites embedded in composite resines used as reinforcement (Hinostroza G., 2006; Sabbagh J. et al (2004).

This study evaluated mechanical properties such as ultimate compressive strengh, hardness and Elasticity modulus for packable resines and cerometers handled according to manufacturer specifications for all materials applying aditional polimerization to packable resines (Guzman H., 2003; Touati B., 1997).

Results confirmed that filling particle size, shape and volume content enhance material´s properties such as hardness and ultimate compressive strengh. In 1997 Condon reported similar results.

Althought morphological characterization could not be done in this study because lackness of a proper imaging microscope, in 2004 Sabbagh J. et al. concluded that spherical particles present in P60 increase their packability and augment the filled volume in resines. The smooth, rounded shape of the filler also enhances material´s fracture strength. However, not all composite resines and ceromers particles are round like P60´s but in an irregular manner and the study states “as mechanical stress tends to concentrate on irregularities of the filler/matrix interface, angles and protuberances of filler particles are zones where cracks initiate promptly”.

P60 showed the highest values for ultimate compressive strenght with a 273.26 MPa. Aditionally the highest VHN were given by P-60 with a 93.62 average. This supports what Da Fonte published in 2004 who states that Z100, being a hybrid resine, as P60 is, showed the highest hardness value (110 VHN in that case) and a ultimate compressive strenght of 275 MPa. P60´s manufacturer (3M) reports a 97,20 ±0,26 VHN (Filtek P60. 3M, 1999), this study provides with close VHN results.

Ceromers are, in theory, materials that show a better mechanical behavior when compared to resines (Touati, 1997). However, results from this study were not conclusive because such mechanical behavior depends on the inorganic matrix material volume, and according to the manufacturer, it varies. Ceromers like BelleGlass (Kerr), Targis (Ivoclar Vivadent) report higher inorganic matrix material volume than Gradia y Adoro (Hopfauf S., 2004). These values are similar to the ones reported for packable resines such as P60.

According to manufacturer IVOCLAR in 2004 (Hopfauf, 2004) reported an inorganic matrix material volume of 35.8 and 40% for Gradia and Adoro, respectively, which are lower than packable resines P60, with 62%, and SureFil at 60%, respectevely (Filtek P60-3M, 1999). However, in 2006 Henostroza citing Henao stated that Adoro - Ivoclar

showed a 75% inorganic matrix material volumen and 64 % for Gradia (Loguercio A., 2006). According to this author, this will make Adoro and Gradia have superior mechanical properties than packable resines.

A heat treatment procedure improves polimerization grade as well as mechanical and phisycal properties (Guzman H., 2003). According to Wendt (1987) a heat treatment improves mechanical properties between 60 to 70% for resines after being photocured especially if they are submitted for five minutes to a temperature between 120 and 123 C. Because of that, it is why packable resines showed a higher ultimate compressive strenght mean with heat treatment (P60-3M: 273.26 MPa and SureFil-Dentsply: 268.96 MPa) when compared to packable resines without heat treatment (P60-3M: 254.51 MPa and SureFil- Dentsply: 242.75 MPa).

2.3. Conclusions

Althought this was not the purpose of the study, mechanical properties results showed that the use of packable resines P-60 (3M) y SureFil (Dentsply) with a double curing technique (lamp and boiling water) offers better mechanical performance.

P-60 showed higher hardness and higher ultimate compressive strenght.

P-60 with aditional heat treatment (seven minutes in boiling water) showed higher hardness and ultimate compressive strenght values than P60 cured with light only.

P60 and Sufefil cured with light and cured with light and heat treatment are the hardest, the highest ultimate comrpessive strenght and the highest Young modulus.

It is seen a big difference in Young modulus when Surefil is not additionally heat treated.

2.4. Recommendations

Conduct other tests in a better in vivo oral–simulated-enviroment (fresh teeth in situ, humidity, chemistry and dynamic cicles). Wear, color, and impact resistance tests will provide a more comprehensive independent evaluation of mechanical and physical properties that reach a real clinic situation.

Following the lines of medical, ethical, and legal procedures a clinical study should be continued to evaluate in-situ performance of the same materials.

3. ACKNOWLEDGEMENTS

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