PONTIFÍCIA UNIVERSIDADE CATÓLICA DO RIO GRANDE DO SUL

Análise de Metodologias de Microdureza aplicadas a Compósitos: é possível comparar resultados utilizando-se diferentes protocolos?

Gustavo Frainer Barbosa –Doutorando em Odontologia, ênfase em Materiais Dentários (PUCRS)

Orientador: Hugo Mitsuo Silva Oshima – Professor Doutor do Departamento de Materiais Dentários (PUCRS)

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RESUMO

Objetivo: Primeiramente, avaliar, em uma resina composta, a correlação existente, em testes de microdureza, entre os fatores carga e tempo de aplicação, utilizando-se os métodos de ensaio mecânico Vickers e Knoop. Em segundo lugar, avaliar, em uma resina composta, os fatores carga e tempo de aplicação no teste de microdureza Knoop, e mostrar, com os resultados obtidos, quando é possível, através dos testes, fazer uma comparação com o grau de conversão da resina composta.

Materiais e Métodos: Resina Grandio (Voco, Cuxhaven, Alemanha), cor A2, foi utilizada para confeccionar as amostras. Noventa (90) amostras foram feitas em uma matriz de politetrafluoretileno com seis (6) mm de diâmetro por três (3) mm de profundidade, onde dois incrementos equidistantes foram fotopolimerizados por 20s cada um através de um dispositivo LED (Celalux, Voco, Cuxhaven, Alemanha, com 800 mW/cm^2). As amostras foram divididas aleatoriamente em três grupos de acordo com o fator carga. Os grupos I, II e III receberam cargas de 50g, 100g e 500g, respectivamente. Estes grupos foram divididos em nove subgrupos de acordo com o fator tempo de aplicação (15s, 30s, 45s). Cada amostra recebeu edentações de um dispositivo Shimadzu HMV tester (Shimadzu, Kioto, Japão). Os resultados foram analisados estatisticamente através de ANOVA com fatores fixos (carga e tempo de aplicação), e ao teste de comparações múltiplas de Tukey ($\alpha = 0.05$).

Resultados: Diferenças significativas foram encontradas entre os grupos para cada metodologia, Vickers e Knoop ($p<0.001$). Entre as amostras testadas com a metodologia Vickers (VHN), a média encontrada variou de 164.94 (50g 45s) até 210.33 (100g 45s). Os valores de microdureza para a metodologia Knoop (KHN) variaram de 128.92 (500g 45s) até 184.26 (100g 15s). Para ambas as metodologias, tanto o fator tempo e o fator tempo de aplicação foram estatisticamente significantes ($p<0.001$), isto mostra que diferentes cargas e tempos de aplicação influenciam na microdureza das resinas.

Conclusão: Primeiramente, este estudo demonstrou que correlacionar os resultados de testes de microdureza Vickers and Knoop não é recomendado, e protocolos similares devem ser aplicados a fim de permitir comparações entre diferentes estudos que utilizam o mesmo tipo de teste. Em Segundo lugar, este estudo demonstrou que os resultados de microdureza Knoop não são recomendados para comparações com o grau de conversão das resinas compostas, e protocolos similares devem ser aplicados a fim de permitir este tipo de correlação.

Key Words: Metodologias. Dureza. Resina composta.

ABSTRACT

Objective: Firstly, to evaluate correlation between load and dwell time in composite resin microhardness tests, using Vickers and Knoop mechanic test methods. Secondly, to correlate time and load on Knoop microhardness test over a composite resin, and to show, with the obtained results, whether composite resin conversion degree comparison is possible through the tests.

Materials and Methods: Resin Grandio (Voco, Cuxhaven, Germany), shade A2, was used to make the samples. . Ninety (90) samples were made on a six-(6)-mm-diameter by three-(3)-mm-deep polytetrafluorethylene matrix, where two equidistant increments were each photocured for 20s by the LED device (Celalux, Voco, Cuxhaven, Germany, with 800 mW/cm^2). The samples were randomly divided into three groups according to the load factor. Groups I, II and III received loads at 50g, 100g and 500g respectively. These groups were divided into nine subgroups according to the deal time (15s, 30s, 45s). Each sample received indentations from a Shimadzu HMV tester (Shimadzu, Kyoto, Japan). The results were submitted to a two-way ANOVA with fixed factors (load and dwell time), and to the post-hoc Tukey multiple comparison test at $\alpha = 0.05$.

Results: Significant differences were recorded between groups for each methodology, Vickers and Knoop ($p<0.001$). Among the Vickers (VHN) tested samples, the average recorded ranged from 164.94 (50g for 45s) to 210.33 (100g for 45s). Knoop microhardness (KHN) ranged from 128.92 (500g 45s) to 184.26 (100g 15s). For both methodologies, both load and dwell time were statistically significant ($p<0.001$), this way showing that different loads and dwell time influence the microhardness of resins.

Conclusion: Firstly, this study demonstrated that correlating both Vickers and Knoop microhardness test results is not recommended, and similar protocols must be applied to allow comparisons among the different studies that use the same kind of test. Secondly, this study demonstrated that Knoop microhardness test results is not recommended for composite resin conversion degree comparisons, and similar protocols should be applied to allow this kind of correlation.

Key Words: Methods. Hardness. Composite resins.

Sumário

1 Apresentação

A dureza é uma propriedade mecânica que pode ser conceituada como a resistência que um material apresenta ao risco ou à formação de uma marca permanente quando pressionado por outro material ou por marcadores padronizados (Garcia, 2000).

Os valores de dureza apresentados por um determinado material possuem uma dependência direta dos tipos de ligações atômicas, iônicas ou moleculares nele existentes. Nos sólidos moleculares, como os plásticos, as forças de Van der Waals atuam entre as moléculas e, por se tratarem de forças baixas, estes materiais são considerados relativamente macios. Já nos sólidos metálicos e iônicos, como a natureza das forças ligantes é mais intensa, esses materiais são mais duros, enquanto os materiais de ligação covalente são, conhecidamente, os materiais de maior dureza (Garcia, 2000).

No século XVIII, teve início a avaliação científica dessa propriedade dos materiais, quando se convencionou, então, que a dureza de um material estava ligada à capacidade que ele apresentava em arranhar outro material. Assim sendo, quanto maior o risco provocado, mais duro era o material. A primeira escala de dureza foi criada pelo mineralogista alemão Friedrich Mohs. Essa escala qualitativa atribui aos materiais um número que indica a sua dureza, sendo o valor 1 atribuído ao talco e o valor 10, valor máximo, ao diamante. A escala de Mohs é utilizada até os dias de hoje como uma referência rápida quanto à dureza dos materiais (Blando, 2001).

Com a necessidade de qualificar e quantificar mais precisamente a dureza de qualquer material, o desenvolvimento de pesquisas sobre o mecanismo dessa propriedade nos materiais acarretou o surgimento de novas técnicas de medida. Dentre as novas técnicas de medida de dureza, as mais difundidas e utilizadas até hoje residem no método de identação (Blando, 2001).

O método identação, ou ensaio de dureza, consiste na aplicação de uma pressão com uma ponta de penetração, a qual irá imprimir uma marca na superfície da peça/material analisado. A medida da dureza será dada em função das características

da marca de impressão e da força aplicada. O primeiro ensaio de identação padronizado e reconhecido industrialmente foi o ensaio de Brinell, proposto pelo metalurgista sueco Johan August Brinell, por volta do ano de 1900. Posteriormente, em 1919, surgiu o teste de Rockwell, criado pelo metalurgista americano Stanley P. Rockwell. Na década de 50, os estudos de Tabor permitiram avanços importantes para os testes de dureza, ao relacionar a curva de descarga com propriedades plásticas e elásticas dos materiais (Blando, 2001).

Os testes de microdureza surgiram, ainda na década de 50, da necessidade de se utilizar cargas muito menores, porque os testes convencionais já não possuíam cargas suficientemente baixas para medir somente a dureza de uma superfície tratada, ou de um filme com espessura pequena (Blando, 2001). Atualmente os testes de microdureza mais difundidos e utilizados são os métodos Vickers e Knoop. Ambos os métodos são bem adequados para a medição de dureza em regiões pequenas e selecionadas dos corpos de prova, sendo o método Knoop indicado para testar materiais frágeis (Callister, 2002).

Uma vez que a Odontologia não é uma ciência ímpar, isolada dentro de um contexto, a utilização destes métodos está presente na constante construção e evolução do conhecimento odontológico e, conseqüentemente, no desenvolvimento de novos materiais e aprimoramento dos materiais já existentes e utilizados no dia a dia do Cirurgião Dentista.

Neste sentido, as resinas compostas têm sido constantemente testadas e analisadas através de ensaios mecânicos *in vitro*. Dentre os inúmeros ensaios mecânicos possíveis de serem realizados, o ensaio de microdureza possui especial destaque, pois é definido como um teste simples para determinar a resistência do material após submetê-lo a edentações (Polydorou *et al*., 2007; Nayif *et al*., 2007). Para Mota *et al*. (2006), a microdureza de um material define sua capacidade de resistir ao desgaste, principalmente, em regiões de áreas funcionais. Poskus *et al*. (2004), Correr *et al*. (2005) e Tango *et al*. (2007), também destacam que os testes de dureza são comumente usados para indicar o grau de conversão nas reações de polimerizações.

Para Poskus *et al*. (2004), existe uma correlação entre os valores de dureza e o grau de conversão dos monômeros após a polimerização. Sendo assim, diversos fatores externos podem influenciar no resultado dos testes de microdureza e, consequentemente, no resultado final da restauração, tais como distância da fonte de luz (Rode *et al*., 2007), o tempo de polimerização das resinas, a densidade de energia (Tango *et al*., 2007). Fatores internos também podem influenciar os resultados obtidos, podemos citar como exemplo a composição (Nayif *et al*., 2007), a cor e a opacidade da resina (Correr *et al*., 2005).

No entanto, possíveis fatores relacionados diretamente aos métodos de microdureza não estão descritos na literatura como sendo passíveis de influenciar nos resultados obtidos. Nos trabalhos que utilizam ensaios mecânicos de microdureza não é possível observar padronização nem na carga aplicada sobre as amostras, nem no tempo que esta carga atua sobre a amostra. Também não há padronização quanto ao tipo de teste de microdureza (Vickers ou Knoop) a ser empregado. Isto pode ser observado em alguns trabalhos científicos descritos no quadro 1.

Portanto, o objetivo deste estudo é comparar os resultados obtidos em testes de microdureza, sobre uma resina composta, quando utilizados diferentes protocolos, e avaliar a possibilidade de se fazer analogias com base nos resultados obtidos.

Tabela 1: Diversificação das variáveis empregadas em testes de microdureza apresentados em artigos científicos.

Artigo	Força	Tempo	Ensaio
Hahnel S, Henrich A, Bürgers R, Handel G, Rosentritt M. **Investigation of Mechanical Properties of Modern Dental Composites After Artificial Aging for One Year.** Oper Dent. 2010; 30(4): 412-419.	500g	60seg	Vickers
Komori PCP, Paula AB, Martin AA, Tango RN, Sinhoreti MAC, Correr-Sobrinho L. **Effect of Light Energy Density on Conversion Degree and Hardness of Dual-cured Resin Cement.** Oper Dent. 2010; 30(5): 120-124.	50g	15seg	Knoop
Camargo EJ, Moreschi E, Baseggio W, Cury JA, Pascotto RC. **Composite depth of cure using four Polymerization techniques.** J Appl Oral Sci. 2009; 17(5):446-50.	25g	5seg	Knoop
Fleming GJP, Awan M, Cooper PR, Sloan AJ. **The potential of a resin-composite to be cured to a 4mm depth.** Sloan. Dental Materials 24 (2008) 522–529.	500g	15seg	Vickers

Aguiar FH, e Oliveria TR, Lima DA, Ambrosano G, Lovadino JR. **Microhardness of different thicknesses of resin composite polymerized by conventional photocuring at different distances**. Gen Dent. 2008 Mar-Apr;56 (2):144-8.	25g	10seg	Knoop
Hannig C, Duong S, Becker K, Brunner E, Attin T. **Effect of bleaching on subsurface micro-hardness of composite and a polyacid modified composite.** Dent Mater. 2007; 23: 198–203.	2N	-	Knoop
David JR, Gomes OM, Gomes JC, Loguercio AD, Reis A. **Effect of exposure time on curing efficiency of polymerizing units equipped with light-emitting diodes**. Journal of Oral Science, Vol. 49, No.1, 19-24,2007.	50g	30seg	Vickers
Aguiar FHB, Braceiro A, Lima DA, Ambrosano GMB, Lovadino JR. **Effect of Light Curing Modes and Light Curing Time on the Microhardness of a Hybrid Composite Resin**. The Journal of Contemporary Dental Practice, Volume 8, No. 6, September 1, 2007.	25g	10seg	Knoop
Polydorou O, Mönting JS, Hellwig E, Auschill TM. **Effect of in-office tooth bleaching on the microhardness of six dental esthetic restorative materials.** Dental Materials 23 (2007) 153–158.	50g	30seg	Knoop
Yazici AR, Kugel G, Gül G. **The Knoop Hardness of a Composite Resin Polymerized with Different Curing Lights and Different Modes.** The Journal of Contemporary Dental Practice, Volume 8, No. 2, February 1, 2007.	500g	15seg	Knoop
Nayif MM, Nakajima M, Aksornmuang J, Ikeda M, Tagami J. **Effect of adhesion to cavity walls on the mechanical properties of resin composites.** Dent Mater. 2008; 24: 83–89.	50g	15seg	Knoop
Tango RN, Sinhoreti MAC, Correr AB, Sobrinho LC, Consani RLX. **Effect of Veneering Materials and Curing Methods on Resin Cement Knoop Hardness.** Braz Dent J. 2007; 18(3): 235-239.	50g	15seg	Knoop
Brandt WC, Moraes RR, Sobrinho LC, Sinhoreti MAC, Consani S. **Effect of different photo-activation methods on push out force, hardness and cross-link density of resin composite restorations.** Dent Mater. 2008; 24: 846-850.	50g	15seg	Knoop
Rode KM, Kawano Y, Turbino ML. **Evaluation of Curing Light Distance on Resin Composite Microhardness and Polymerization.** Oper Dent. 2007; 32-6: 571-578.	50g	45seg	Vickers
Mota EG, Oshima HMS, LHB, Pires LAG, Rosa RS. **Evaluation of diametral tensile strength and knoop microhardness of five nanofilled composites in dentin and enamel shades.** Stomatologija. 2006; 8:67-9.	100g	15seg	Knoop
Correr AB, Sinhoreti MAC, Sobrinho LC, Tango RN, Schneider LFJ, Consani S. **Effect of the Increase of Energy Density on Knoop Hardness of Dental Composites Light-Cured by Conventional QTH, LED and Xenon Plasma Arc.** Braz Dent J. 2005; 16(3): 218-224.	50g	15seg	Knoop
Poskus LT, Placido E, Cardoso PEC. **Influence of placement techniques on Vickers and Knoop hardness of class II composite resin restorations.** Dent Mater. 2004; 20: 726–732.	100g	15seg	Knoop
Neves AD, Discacciati JAC, Oréfice RL, Jansen WC. **Correlation between degree of conversion, microhardness and inorganic content in composites**. Pesqui Odontol Bras. 2002; 16 (4): 349-354.	200g	15seg	Vickers

2 Artigo Submetido ao Journal of Applied Oral Science

Analysis of microhardness methodologies applied to composites: is it possible to compare results using different protocols?

Objective: To evaluate correlation between load and dwell time in composite resin microhardness tests, using Vickers and Knoop mechanic test methods.

Materials and Methods: Resin Grandio (Voco, Cuxhaven, Germany), shade A2, was used to make the samples. Ninety (90) samples were made on a six-(6)-mm-diameter by three-(3)-mm-deep polytetrafluorethylene matrix, where two equidistant increments were each photocured for 20s by the LED device (Celalux, Voco, Cuxhaven, Germany, with 800 mW/cm^2). The samples were randomly divided into three groups according to the load factor. Groups I, II and III received loads at 50g, 100g and 500g respectively. These groups were divided into nine subgroups according to the deal time (15s, 30s, 45s). Then, the light exposed surface of each composite resin sample was divided into two hemispheres. Each sample side received three indentations, totaling 540 indentations with Shimadzu HMV tester (Shimadzu, Kyoto, Japan). The Vickers (VHN) and Knoop (KHN) results were submitted to a two-way ANOVA with fixed factors (load and dwell time), and to the post-hoc Tukey multiple comparison test at $\alpha = 0.05$.

Results: Significant differences were recorded between groups for each methodology, Vickers and Knoop ($p<0.001$). Among the Vickers (VHN) tested samples, the average recorded ranged from 164.94 (50g for 45s) to 210.33 (100g for 45s). Knoop microhardness (KHN) ranged from 128.92 (500g 45s) to 184.26 (100g 15s). For both methodologies, both load and dwell time were statistically significant ($p<0.001$), this way showing that different loads and dwell time influence the microhardness of resins.

Conclusion: This study demonstrated that correlating both Vickers and Knoop microhardness test results is not recommended, and similar protocols must be applied to allow comparisons among the different studies that use the same kind of test.

Key Words: Methods. Hardness. Composite resins.

INTRODUCTION

Mechanical tests are commonly used to check-up and improve several classes of dental materials. One of such commonly used tests on dentistry research is the hardness test. Hardness can be defined as the resistance of a material to indentation [16]. Moreover, hardness is related to the material strength, its proportional limit and its ability to abrade or to be abraded by opposing dental structures/materials[16].

Development of materials, such as composite resins, filled with smaller particles, has been increasingly using microhardness testers that use less than 1 Kgf during the indentation. Historically, Vickers and Knoop microhardness tests have been used by the majority of investigators for testing hardness of this kind of material [3-5,7,8,10,12-17,19,21,22,24,26]. This can be explained by the fact that such test is a simple and reliable method [14] to show the strength of materials through their resistance to indentation.

Although microhardness tests are widely used tests, the measured hardness depends on test load and dwell times [21]. However, there is no standardization neither on the load applied value, nor on the time of load application on the composite sample. Also, correlations between Vickers and Knoop microhardness tests are commonly carried out, but no work to date has related that possibility and there may be no correlation in hardness values when different indenter shapes are compared [24].

Thus, the aim of this *in vitro* study was to correlate time and load of each kind of microhardness test (Vickers / Knoop) on a composite resin and then show, with the obtained results, if comparison between these two different microhardness tests is possible.

MATERIALS AND METHODS

A commercially available nanohybrid composite resin (Grandio, Voco, Cuxhaven, Germany, Lot 732242), shade A2, was used to prepare 90 samples. These samples were made using a PTFE (polytetrafluorethylene) 6 mm diameter and 3 mm high double entry mold. The PTFE mold was placed on a glass plate and the composite resin was inserted in two 2-mm-thick-maximum increments. To achieve a smooth surface on the last increment, a polyester strip was used on the increment and pressed by means of a glass plate. Each increment was photocured using a LED device (Celalux, Voco, Cuxhaven, Germany, with 800 mW/cm^2) during 20s. The light source power was checked with a radiometer (Demetron, Kerr, Orange, CA, EUA) every five exposures.

The samples were stored in a recipient with distilled water for 24h and protected from light at 37 °C in a culture stove (model 002 CB, Fanem Ltda, São Paulo, SP, Brazil). Afterwards,

the samples were randomly divided into three groups according to the load factor. Groups I, II and III received loads at 50g, 100g and 500g respectively. These groups were divided into nine subgroups according to the deal time (15s, 30s, 45s) as shown on Table 1.

Then, the light exposed surface of each composite resin sample was divided into two hemispheres. Each sample side received three indentations, totaling 540 indentations with Shimadzu HMV tester (Shimadzu, Kyoto, Japan). The Vickers (VHN) and Knoop (KHN) results were submitted to a two-way ANOVA with fixed factors (load and dwell time), and to the post-hoc Tukey multiple comparison test at $\alpha = 0.05$.

RESULTS

Significant differences were recorded between groups for each methodology, Vickers and Knoop ($p<0.001$). Among the Vickers (VHN) tested samples, the average recorded ranged from 164.94 (50g for 45s) to 210.33 (100g for 45s). Knoop microhardness (KHN) ranged from 128.92 (500g 45s) to 184.26 (100g 15s). Multiple comparisons are presented on Tables 2, 3, 4 and on Graphs 1, 2. For both methodologies, both load and dwell time were statistically significant ($p<0.001$).

DISCUSSION

The Vickers and Knoop hardness tests have been used for analysis of composite resin conversion degree in several studies [5,7,9,15,19]. These studies advocate that higher obtained values, through microhardness tests, correspond to higher composite resin conversion degree, obtained by photo-polymerization [7,19]. Recent studies have used different dwell times and different load values to analyze this conversion. However, a direct analogy cannot be drawn, because there are significant differences between these methods and between the variables used [21]. Some of these existing variables are a result of internal factors (such as filler, particle size, polymeric matrix), and of external factors (such as distance from the light power source, time of composite resin polymerization, energy density, composition, shade and resin composite opacity) that may influence on the microhardness tests.

Another existing variable is the type of light power source. Different light units result in conversion value variations [6,7,15,18,19,20,23], what can be explained by the existing characteristics of each light source, such as light bulb type, number and disposition plus power and heat produced inside the light source units. In a specific study [7], three different light power sources (LED, PAC and quartz-tungsten-halogen light) were used on two different composite resins. The

hardness obtained from the composites photocured by PAC source was statistically lower when compared with the composites photocured by LED or by the halogen light. But there is no difference between these two last light sources, independently from the resin increment thickness. Regarding dwell time, it was observed that the increase in time exposure increased Knoop hardeness values in the composite resin when LED or PAC was used. As to the halogen light, the increase in dwell time did not influence the Knoop hardness values, but these values decreased as the resin increment thickness increased. Besides this, it was demonstrated that the composite resin increment size can modify the polymerization degree [4]. According to Aguiar, et al.[4]. (2007), the polymerization method is a determinant factor and the manufacturer's recommended time for polymerizing a composite resin is not enough for an "ideal polymerization".

For these reasons, this study used the same light source (LED), at the same distance, with the same polymerization time (20s) and same dwell time on all samples. Since 2-mm-thick-maximum increments were used for each polymerization, the thickness variant of the increment did not act in this study, this way satisfying the works by David, et al.[8]. (2007), Aguiar, et al.[4]. (2007). Following preparation, the samples were tested for Knoop and Vickers loads with different dwell times. Significant non-linear differences among them were noted, demonstrating that different loads and dwell times influence the resin microhardness, according to what Shahdad et al.[21]. (2007) have stated, that is, the variation of hardness with load is a well-known artifact of traditional hardness testing and is often known as the indentation size effect.

In this way, the strong correlation that some studies claim to exist between wear resistance and hardness, as it is shown at Abe, et al.[2]. (2001) in their study, in which they found a strong correlation between wear resistance and Knoop hardness of the materials tested, may not exist. Other studies have either completely ruled it out or suggested a limited correlation between the hardness and wear resistance [1,11,25]. This correlation between wear resistance and hardness can only be possible if the same testing protocol is applied, and the obtained results are compared to different polymerization time.

To Shahdad et al.[21]. (2007) Vickers and Knoop hardness tests seem to be the preferred choice of test among majority of the investigators. However, the comparisons could be inappropriate, because there is a great deal of variability among the tests that have been performed. And besides, these tests have a specific limitation that is the microscopic measurements of hardness indentations after the indenter is removed. These measurements can be affected by some factors, like as, the limitation in resolution of the optical system, the perception of the operator and the elastic recovery of the material

Therefore, for the hardness test analysis to be faithful to both Knoop and Vickers, a universal rule for future studies is needed, with equal dwell time and load values. This way, the odds of loyalty of further studies will be greater. Moreover, direct comparisons between the two different types of mechanical test, cited above, are not viable as show by the majority of the obtained results where VHN are greater than KHN. Thus, we see that past studies 'could not have made comparisons' among different times and loads, because there are differences between the tests and, consequently, in their results, making any comparative statement unworkable.

CONCLUSION

This study demonstrated that correlating both, Vickers and Knoop, microhardness test results is not recommended, and similar protocols must be applied to allow comparisons among the different studies that use the same kind of test.

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Table 1: The random division has created 9 groups.

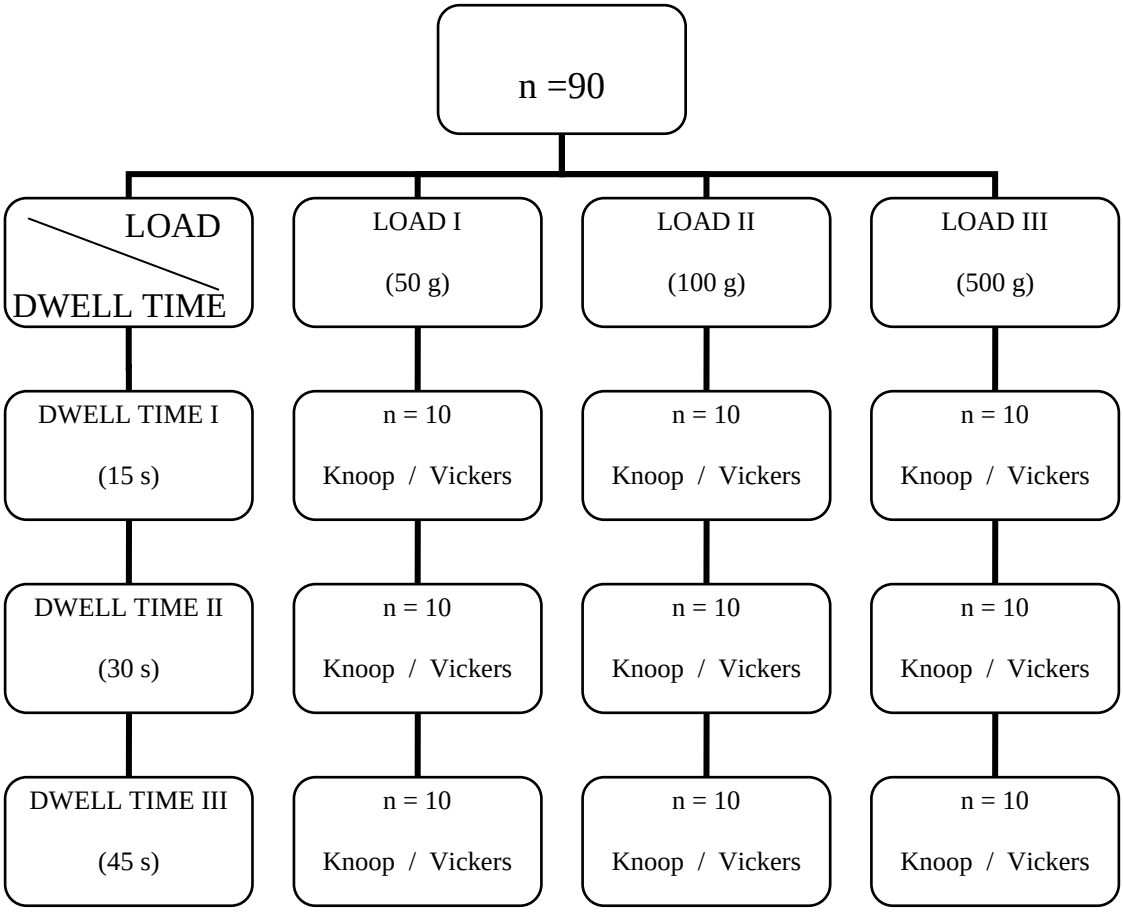


Table 2: Means comparison for all groups.

	50g		**100g**		**500g**	
	VHN	KHN	VHN	KHN	VHN	KHN
15s	179.32	168.13	202.04	184.25	181,58	140.77
30s	185.74	149.87	197.20	179.13	193.76	139.40
45s	164.94	180.33	210.33	148.09	186.56	128.92

Table 3: Means for Vickers groups in homogeneous subsets are displayed.

Tukey HSD[a]

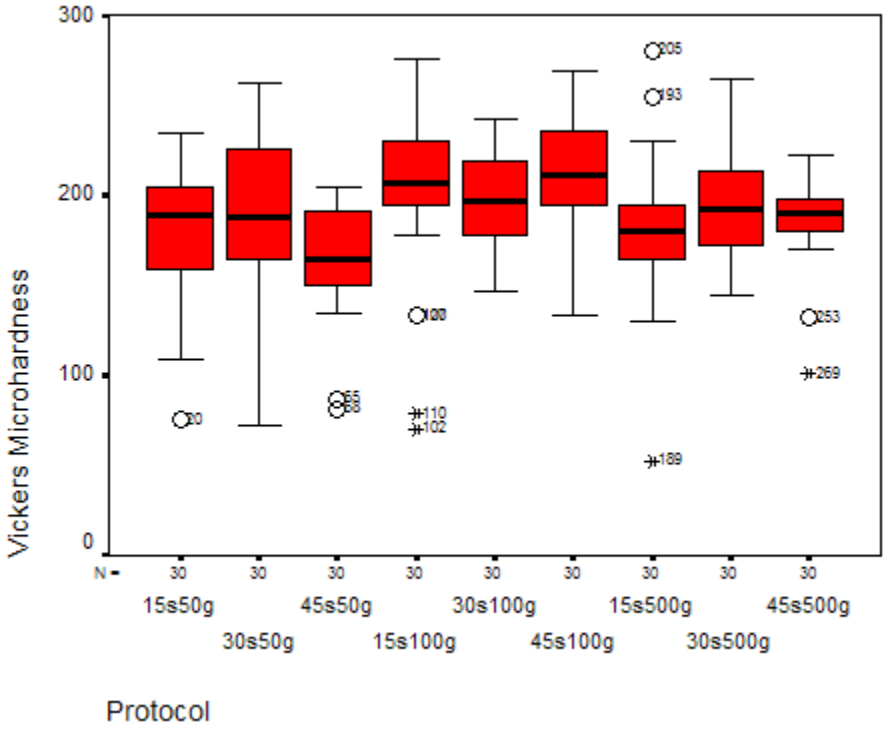
GROUP	N	Subset for alpha = .05		
		1	2	3
45s50g	30	164,9467		
15s50g	30	179,3233	179,3233	
15s500g	30	181,5867	181,5867	
30s50g	30	185,7433	185,7433	185,7433
45s500g	30	186,5667	186,5667	186,5667
30s500g	30		193,7667	193,7667
30s100g	30		197,2000	197,2000
15s100g	30		202,0467	202,0467
45s100g	30			210,3333
Sig.		,282	,220	,136

Table 4: Means for Knoop groups in homogeneous subsets are displayed.

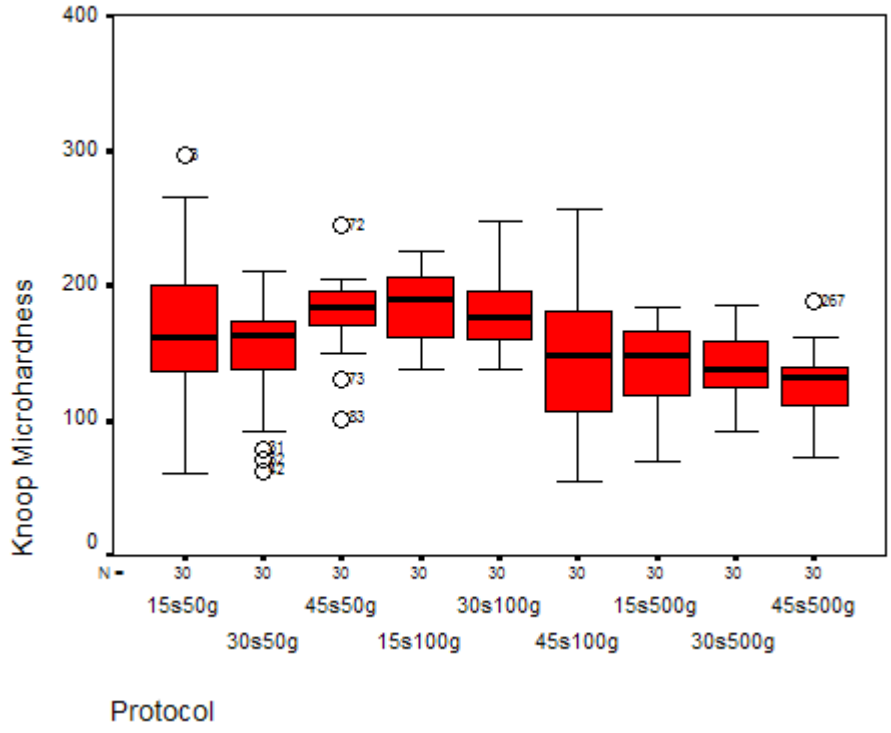
Tukey HSD[a]

GROUP	N	Subset for alpha = .05		
		1	2	3
45s500g	30	128,9200		
30s500g	30	139,4067		
15s500g	30	140,7767	140,7767	
45s100g	30	148,0900	148,0900	
30s50g	30	149,8700	149,8700	
15s50g	30		168,1333	168,1333
30s100g	30			179,1333
45s50g	30			180,3333
15s100g	30			184,2667
Sig.		,350	,070	,708

Graph 1: Means and outliers in Vickers Microhardness results.



Graph 2: Means and outliers in Knoop Microhardness results.



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3 Artigo Submetido à Operative Dentistry

Analysis of Knoop microhardness test: is it possible to make an analogy between conversion degree and the obtained results using different protocols?

Objective: To correlate time and load on Knoop microhardness test over a composite resin and to show, with the obtained results, whether composite resin conversion degree comparison is possible through the tests.

Materials and Methods: Resin Grandio (Voco, Cuxhaven, Germany), shade A2, was used to make the samples. Ninety (90) samples were made on a six-(6)-mm-diameter by three-(3)-mm-deep polytetrafluorethylene matrix, where two equidistant increments were each photocured for 20s by the LED device (Celalux, Voco, Cuxhaven, Germany, with 800 mW/cm^2). The samples were randomly divided into three groups according to the load factor. Groups I, II and III received loads at 50g, 100g and 500g respectively. These groups were divided into nine subgroups according to the deal time (15s, 30s, 45s). Each sample received three indentations, totaling 270 indentations with Shimadzu HMV tester (Shimadzu, Kyoto, Japan). The Knoop (KHN) results were submitted to a two-way ANOVA with fixed factors (load and dwell time), and to the post-hoc Tukey multiple comparison test at $\alpha = 0.05$.

Results: Significant differences were recorded between groups. Among the Knoop microhardness (KHN) tested samples, the average recorded ranged from 128.92 (500g 45s) to 184.26 (100g 15s). For this methodology, both load and dwell time were statistically significant ($p<0.001$), this way showing that different loads and dwell time influence the microhardness of resins.

Conclusion: This study demonstrated that Knoop microhardness test results is not recommended for composite resin conversion degree comparisons, and similar protocols should be applied to allow this kind of correlation.

Running (short) title: Analysis of Knoop microhardness test

INTRODUCTION

Composite resins are widely used in restorative dentistry. Patients' constant demands for esthetic associated with the enhancement of mechanical properties have brought important improvements to composite resins, and as a result, this restorative material has been given universal applicability. On the posterior restorations, for example, the material is constantly under masticatory stresses. So, composite resins with good mechanical properties should be selected for that purpose.[1]

Thus, in order to check up the mechanical properties of this class of restorative materials, mechanic tests have been commonly used, being the so-called hardness tests one of them. Such test is a simple and reliable method to reflect the strength of a given material through its resistance to indentation. Several studies have used the Knoop microhardness test for analysis of the composite resin conversion degree.[2,3,4,5,6] These studies advocate that higher obtained values, through microhardness tests, correspond to higher composite resin conversion degree, obtained by photo-polymerization. [3,6]

However, although correlations between Knoop microhardness tests are commonly performed to compare the composite resin conversion degree, there has not been any work to date that has reported such possibility. Also, the measured hardness depends on load and dwell times test, [7] but there is no standardization neither on the load applied value, nor on the time of load application over the composite resin sample in the published studies.

Therefore, the aim of this *in vitro* study was to correlate time and load on Knoop microhardness test over a composite resin and to show whether an analogy between the composite resin conversion degree and the obtained results is possible.

MATERIALS AND METHODS

A commercially available nanohybrid composite resin (Grandio, Voco, Cuxhaven, Germany, Lot 732242), shade A2, was used to prepare 90 samples. These samples were made using a PTFE (polytetrafluorethylene) 6 mm diameter and 3 mm high double entry mold. The PTFE mold was placed on a glass plate and the composite resin was inserted in two 2-mm-thick-maximum increments. To achieve a smooth surface on the last increment, a polyester strip was used on the increment and pressed by means of a glass plate. Each increment was photocured using a LED device (Celalux, Voco, Cuxhaven, Germany, with 800 mW/cm^2) during 20s. The light source power was checked with a radiometer (Demetron, Kerr, Orange, CA, EUA) every five exposures.

The samples were stored in a recipient with distilled water for 24h and protected from light at 37 °C in a culture stove (model 002 CB, Fanem Ltda, São Paulo, SP, Brazil). Afterwards, samples were randomly divided into 9 groups according to load (50, 100 or 500g) and dwell time (15, 30 or 45s) as shown on Table 1.

Each light exposed surface sample side received three indentations, totaling 270 indentations with Shimadzu HMV tester (Shimadzu, Kyoto, Japan). The Knoop (KHN) results were submitted to a two-way ANOVA with fixed factors (load and dwell time), and to the post-hoc Tukey multiple comparison test at $\alpha = 0.05$.

RESULTS

Significant differences were recorded between groups. Among the Knoop microhardness (KHN) tested samples, the average recorded ranged from 128.92 (500g 45s) to 184.26 (100g 15s). For this methodology, both load and dwell time were statistically significant ($p<0.001$), this way showing that different loads and dwell time influence the microhardness of resins. Multiple comparisons are presented on Tables 1 and 2.

DISCUSSION

The Knoop microhardness test has been shown as one of the best methods for testing the hardness of composite resins [8], and hardness has been shown to be an indirect measure of the degree of conversion.[9] Over the last few years, several studies related to the degree of conversion and mechanical properties of composite resins have been developed.[1] These studies advocate that the radiant exposure generated by the light source can influence the degree of conversion of composite resins and thereby influence their mechanical properties.[1] So, higher obtained values through microhardness tests correspond to higher composite resin conversion degree, obtained by photo-polymerization. [3,6]

Recent studies have used different dwell times and different load values to analyze this conversion on several composite resin brands. However, a direct analogy cannot be drawn, because there are significant differences between these methods and between the variables used.[7] Besides, since the microhardness value is greatly influenced by some variables, a cross-comparison between the different brands is limited.[10] Some of these existing variables are a result of internal factors (such as filler, particle size, polymeric matrix), and of external factors (such as distance from the light power source, time of composite resin polymerization, energy

density, composition, shade and resin composite opacity) that may influence on the microhardness tests.

Another existing variable is the type of light power source. Different light units result in conversion value variations [3,5,6,11,12,13,14], what can be explained by the existing characteristics of each light source, such as light bulb type, number and disposition plus power and heat produced inside the light source units. The polymerization method is a determinant factor, but the manufacturer's recommended time for polymerizing a composite resin is not enough for an "ideal polymerization". [15] Besides this, it was demonstrated that the composite resin increment size can modify the polymerization degree. [15]

For these reasons, this study used the same light source (LED), at the same distance, with the same polymerization time (20s) and same dwell time on all samples. Since 2-mm-thick-maximum increments were used for each polymerization, the thickness variant of the increment did not act in this study. Following preparation, the samples were tested for Knoop loads with different dwell times. Significant non-linear differences among them were noted, demonstrating that different loads and dwell times influence the resin microhardness. The variation of hardness with load is a well-known artifact of traditional hardness testing and is often known as the indentation size effect. [7]

In this way, the strong correlation that some studies claim to exist between conversion degree and hardness may not exist. Other studies have either completely ruled it out or suggested a limited correlation between the hardness and wear resistance [16,17,18]. This correlation between wear resistance and hardness can only be possible if the same testing protocol is applied, and the obtained results are compared to different polymerization time.

Although Knoop hardness tests seem to be the preferred choice of test among the majority of the investigators, the comparisons could be inappropriate, because the hardness values were often greater at the center of the specimens.[8] And besides, these tests have a specific limitation that is the microscopic measurements of hardness indentations after the indenter is removed. These measurements can be affected by some factors such as the limitation in resolution of the optical system, the perception of the operator and the elastic recovery of the material.

Therefore, for the Knoop microhardness test analysis to be faithful, a universal rule for future studies is needed, with equal dwell time and load values. This way, the odds of loyalty of further studies will be greater. Moreover, direct comparisons between hardness and composite resin conversion degree, are not viable. Thus, we see that past studies 'could not have made analogies between conversion degree and Knoop microhardness tests' among different times

and loads, because there are differences between the tests and, consequently, in their results, making any comparative statement unworkable.

CONCLUSION

This study demonstrated that Knoop microhardness test results are not recommended to composite resin conversion degree comparisons, and similar protocols should be applied to allow this kind of correlation.

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Table 1: Means comparison for all groups.

	50g	**100g**	**500g**
	KHN	KHN	KHN
15s	168.13	184.25	140.77
30s	149.87	179.13	139.40
45s	180.33	148.09	128.92

Table 2: Means for Knoop groups in homogeneous subsets are displayed.

Tukey HSD[a]

GROUP	N	Subset for alpha = .05		
		1	2	3
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15s500g	30	140,7767	140,7767	
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30s50g	30	149,8700	149,8700	
15s50g	30		168,1333	168,1333
30s100g	30			179,1333
45s50g	30			180,3333
15s100g	30			184,2667
Sig.		,350	,070	,708

4 Discussão Geral

Os métodos de ensaio mecânico Vickers e Knoop têm sido utilizados para avaliar as propriedades mecânicas dos materiais em diversos estudos. Na odontologia a utilização destes testes é muito comum, sendo a resina composta um dos materiais de uso odontológico que, frequentemente, recebe este tipo de análise. Uma propriedade mecânica constantemente avaliada neste material é o grau de conversão obtido com a fotoativação. Quanto maior for o tempo de fotoativação maior será o grau de conversão alcançado. Desta forma, através dos resultados obtidos, é feita uma analogia que cria uma relação direta entre o valor encontrado e o grau de conversão da resina composta. Assim sendo, quanto maior forem os valores obtidos através dos testes de microdureza, maior será, portanto, o grau de conversão alcançado com a fotoativação das resinas compostas. Estudos recentes utilizaram diferentes tempos de aplicação e diferentes valores de carga para avaliar esta conversão, como pode ser verificado através dos trabalhos citados nos artigos apresentados e, também, através da Tabela 1, presente na apresentação deste trabalho. Porém, existem diferenças significativas entre estes estudos e entre as variáveis utilizadas que impossibilitam uma analogia direta dos seus resultados. Um exemplo destas diferenças e/ou variáveis existentes é a presença de fatores internos presentes em uma resina composta, tais como, o tipo de carga, o tamanho da partícula e a matriz polimérica, que podem influenciar os resultados alcançados. Segundo Yan *et al*. (2010), o valor de microdureza é fortemente influenciado por fatores, tais como a carga das partículas que compõe o compósito e, por este motivo, comparações entre diferentes marcas comerciais são limitadas. Conforme Hahnel *et al*. (2010), existe um número considerável de materiais compósitos no que tange o tipo de partícula empregada. Todavia, diferenças entre estes materiais referentes ao tipo do sistema monomérico, ao tipo de composição das partículas e ao tipo de união química das partículas matriciais podem contabilizar performances mecânicas diferentes e podem ocasionar diferenças na resistência dos materiais à degradação química e mecânica.

Existem, também, fatores externos, tais como a distância da fonte de luz, o tempo de polimerização das resinas, a densidade de energia, a composição, a cor e opacidade da resina que também podem influenciar nos testes de microdureza.

Para Fróes-Salgado *et al*. (2009), quando a distância entre o compósito e a fonte de luz aumenta uma severa atenuação da luz é observada. Segundo Neves *et al*. (2002) as diferentes unidades de luz resultam em variações nos valores de conversão, por apresentarem diferenças em suas características, como tipo, número e disposicão das lâmpadas, potência e calor gerado no interior dos aparelhos. Aguiar *et al.* (2007) relataram que o método de polimerização é um fator determinante e que o tempo recomendado pelo fabricante dos compósitos resinosos são insuficientes para uma "ideal polimerização". Para Correr *et al.* (2005) que analisaram diferentes fontes de luz (LED, PAC, e Luz Halógena) em duas resinas diferentes, a dureza dos compósitos fotoativados por PAC foi estatisticamente inferior em relação aos compósitos fotoativados com luz halógena ou LED, que por sua vez, não se diferenciaram entre si, independente da profundidade. Já em relação ao tempo de exposição, constatou-se que o aumento do tempo produziu compósitos com maiores valores de dureza Knoop quando se utilizou LED ou PAC. Para Luz Halógena o aumento de tempo de exposição não influenciou os valores de dureza e que os valores de dureza Knoop diminuíram com o aumento da profundidade. Além disto, Aguiar *et al*. (2007) constataram que o tamanho do incremento de resina também pode modificar o grau de polimerização, o que vai ao encontro do que é afirmado por Camargo *et al.* (2009) e por Pollington *et al*. (2009), que preconizam que o incremento de resina possua no máximo 2mm de espessura.

Por essas razões o presente estudo utilizou a mesma fonte de luz (LED), com a mesma distância, com o mesmo tempo de polimerização (20s) e com o mesmo tempo de aplicação de carga sobre todas as amostras. Assim como, foram realizados incrementos com no máximo 2mm para cada fotoativação, para que esta variante não atuasse neste trabalho, indo, desta forma, ao encontro dos trabalhos de Correr *et al*. (2005), David *et al.* (2007), Aguiar *et al*. (2007). Após a confecção das amostras, estas foram submetidas aos testes Knoop e Vickers com diferentes tempos e cargas constatando-se diferenças significativas e não lineares entre os resultados obtidos,

demonstrando, assim, que diferentes cargas e tempos influenciam na microdureza das resinas, contrariando Shahdad *et al*. (2007) que afirmam que a microdureza independe da carga.

Portanto, para que a análise dos testes de microdureza seja fiel, tanto para o ensaio de microdureza Knoop, quanto para o ensaio de microdureza Vickers, necessita-se de uma regra universal para os futuros estudos e se faz necessário um protocolo de testagem com tempo e carga únicos e iguais, porque, desta maneira, as chances de fidelidade dos futuros estudos serão maiores. Além disto, possíveis comparações diretas entre os dois diferentes tipos de ensaio mecânico, citados anteriormente, se mostraram inviáveis. Variações significativas e não lineares também foram encontradas quando os dois tipos de ensaio foram comparados. Os valores encontrados para os testes realizados com a metodologia Vickers foram, na quase totalidade das vezes, maiores que os valores encontrados quando utilizada a metodologia Knoop. . Desta forma, analogias e comparações entre estes dois tipos de ensaio mecânico devem ser evitados. Assim sendo, podemos perceber que os estudos passados “não poderiam fazer comparações” quando utilizados tempos e cargas diferentes, pois existem diferenças entre os testes realizados e, consequentemente, em seus resultados, ficando, desta forma, qualquer afirmação comparativa inviável de ser realizada.

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ANEXO 1 – Carta de aprovação da Comissão Científica e de Ética da Faculdade de Odontologia da PUCRS

Comissão Científica e de Ética
Faculdade da Odontologia da PUCRS

Porto Alegre 06 de Setembro de 2010

O Projeto de: Tese

Protocolado sob nº:	0035/08
Intitulado:	Análise de metodologias de microdureza aplicadas a compósitos: é possível comparar resultados utilizando-se diferentes protocolos?
Pesquisador Responsável:	Prof. Dr. Hugo Mitsuo Silva Oshima
Pesquisadores Associados	Gustavo Frainer Barbosa, Eduardo Gonçalves Mota
Nível:	Tese / Doutorado

Foi ***aprovado*** pela Comissão Científica e de Ética da Faculdade de Odontologia da PUCRS em *06 de Setembro de 2010.*

Profa. Dra. Ana Maria Spohr
Presidente da Comissão Científica e de Ética da
Faculdade de Odontologia da PUCRS

Av. Ipiranga, 6681, Prédio 06 sala 210
Porto Alegre /RS – Brasil – Cx. Postal:1429
90619-900
Fone/Fax: (51) 3320-3538
e-mail: odontologia-pg@pucrs.br

ANEXO 2 – E-mail de confirmação de submissão de trabalho ao Journal of Applied Oral Science

[JAOS] JAOS-1901 Submission Acknowledgement
Quinta-feira, 16 de Setembro de 2010 13:48
De:
"Carlos F. Santos" <jaos@usp.br>
Adicionar remetente à lista de contatos
Para:
"Gustavo Frainer Barbosa" <gfraibar@yahoo.com.br>

Dear Dr. Gustavo Frainer Barbosa,

Thank you for submitting the manuscript, "JAOS-1901 - Analysis of microhardness methodologies applied to composites: is it possible to compare results using different protocols?" to Journal of Applied Oral Science. With the online journal management system that we are using, you will be able to track its progress through the editorial process by logging
in to the journal web site:

Manuscript URL:
http://submission.scielo.br/index.php/jaos/author/submission/40525
Username: gfraibar

If you have any questions, please contact me. Thank you for considering this journal as a venue for your work.

Yours sincerely,

_____

Carlos F. Santos, DDS, MSc, PhD, Associate Professor
Editor-in-Chief
Journal of Applied Oral Science
http://www.scielo.br/jaos

ANEXO 3 – E-mail de confirmação de submissão de trabalho a Operative Dentistry

6 10-319-L Manuscript received - Operative Dentistry

Terça-feira, 26 de Outubro de 2010 13:59

De:

"editor@jopdent.org" <editor@jopdent.org>
Adicionar remetente à lista de contatos

Para:

gfraibar@yahoo.com.br

Cc:

hoshima@terra.com.br, ed_mota@terra.com.br, anaspohr@terra.com.br, lucianahirakata@yahoo.com.br, marcelfarret@yahoo.com.br

Dear Mr. Barbosa,

On October 26, 2010, I received your manuscript entitled "Analysis of Knoop microhardness test: is it possible to make an analogy between conversion degree and the obtained results using different protocols?" by Gustavo Barbosa, Hugo Oshima, Eduardo Gonçalves, Ana Maria Spohr, Luciana Hirakata, and Marcel Farret.

Your manuscript has been assigned the Paper #: 10-319-L.

You may check on the status of this manuscript by visiting your author home page at http://jopdent.allentrack.net.

Thank you for submitting your work to Operative Dentistry.

Sincerely,

Kevin Matis
Editorial Assistant
Operative Dentistry