The Influence of Testing Protocols on Microhardness Tests of Composite Resin with Different Viscosities

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Abstract

This study tested two hypotheses. The first null hypothesis affirms that there is no statistical difference when composite resin is submitted to knoop or vickers microhardness using different testing protocols. The second hypothesis tested the possible influence of elastic modulus of composite resin in microhardness tests. Two nanohybrid composites were used with different viscosity behavior (Grandio and Grandio Flow). Samples (n=540) were made with 4 mm diameter and 2 mm high using a PTFE mould, stored for 24h at 37°C protected from light and randomly divided into 36 groups according to the resin viscosity (medium and low), microhardness methodology (vickers or knoop), load (50, 100 or 500g) and time (15, 30 or 45s). Two readings were made at the light exposed surface of each sample with Shimadzu HMV tester accomplishing 1.080 indentations. The vickers and knoop results were submitted to a three-way ANOVA with fixed factors the composite viscosity, load and time, and the post-hoc Tukey multiple comparison test at α=0.05. Significant differences were recorded ($p<0.001$) between the microhradness methodologies with two viscosities rejecting both hypotheses rejecting both null hypotheses. Among the vickers tested samples, the average recorded ranged from 164.94 (50g for 45s) to 210.33 (100g for 45s) for the medium viscosity composite and from 31.2 (100g for 30s) to 88 (500g for 45s) for a low viscosity resin. Knoop microhardness results ranged from 128.92 (500g for 45s) to 184.26 (100g for 15s) for medium viscosity composite and from 23.6 (100g for 15s) to 73.4 (500g for 30s) for flowable compositive. In a controlled in vitro experiment, the microhardness formulas mislead researchers to associate an improvement of diagonal measurements to higher load with a linear and proportional behavior. It is not recommended to clinicians compare both vickers and knoop microhardness results of composite materials without similar protocols applied in the research.

Key Words: Microhardness, Resin composite, Dental materials

Introduction

Following the high esthetic expectations of the patients, composite resin improved it's popularity in clinical dentistry, composition and mechanical properties. The composites can be classified by several properties, one of which is viscosity. The viscosity is the property of the corresponding transport of microscopic amount of movement by molecular diffusion and is a measure of resistance to flow of fluid non-crystalline materials [1]. The larger the constituent molecules of a fluid and stronger the intermolecular couplings, the lower its flow and, therefore, the higher its viscosity. The resins can be classified into resins of low, medium and high viscosity. The viscosity of the liquid decreases with increasing temperature and depends on the nature of the substances [1,2].

From 1966, the low viscosity and high fluidity [3] resins emerged with the aim of supplying the necessary features to prevent microleakage, decreasing causes microfractures at the bonded line and favoring marginal sealing. These types of flowable composites have the same type of filler particles with the traditional resins, however in lower weigth %, thereby reducing the viscosity of the mixture to flow, and adapt easily fill the interior angles of the cavity preparation. However, they have lower wear resistance compared to conventional resins which have a greater amount of load and are less susceptible to wear [4,5].

In materials science, hardness is the characteristic property of a solid material expressing its resistance to permanent deformation and is directly related to the bond strength of atoms [1,6,7]. Examines the ability of materials to be edentulous, as an indirect way of understanding the behavior of wear. Its applications range from dental hard tissues, ceramics, alloys up molding materials [8]. The hardness measurement is represented as the ratio of load area where the load is applied. The instrument of load application can have different shapes like sphere, simple elongated pyramidal base and among others, also the area of brand impression is important as the hit value [9,10]. The hardness testers under 1kgf are used during indentations due to changes in testing techniques and materials with increasingly smaller particles. Basically two different methods of analysis are used for microhardness testing restorative materials: Vickers and Knoop [11,12]. Vickers hardness is a classification method based on the hardness of a laboratory test materials [13-15]. In this method, used is a pyramid of diamond with a dihedral angle of 136 which is compressed with an arbitrary force "F" to the surface of the material. Calculate the area "A" printed by measuring its diagonal surface, the fragile material is applied [16-19]. Knoop However, with a ratio between the diagonal 7-1, is used to test the behavior of a flexible material able to shrink the smaller diagonal after removing the load [17,20-22].

Composite filler evolved from macrosized (100 µm) to nanosized (5nm) during last decades. So hardness methodologies were submitted to smaller areas, leading to microhardness. However, different test protocols are recorded in literature and simple and direct comparisons are made [6-22] (*Table 1*). Thus, this research presents as null hypothesis

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Table 1. *A comparison of different test protocols described at the literature.*

Authors	Published year	Load	Impression time	Methodology
Andrade, Basting, Rodrigues, Amaral, Turssi and França.	2014	50g	15s	Knoop
Sabatini	2013	300g	15s	Vickers
Algahtani	2012	300g	15s	Vickers
Catelan, Santo, Menegazzo, Moraes, dos Santos	2012	50g	15s	Knoop
Erdemir	2012	200g	15s	Vickers
Soares-Geraldo	2011	50g	45s	Vickers
Price, Fahey and Felix	2010	50g	10s	Knoop
Correa, Henn, Marimon, Rodrigues and Demarco	2010	50g	30s	Knoop
Voltarelli, dos Santos-Daroz, Alves, Peris and Marchi	2009	25g	20s	Knoop
Fleming, Awan, Cooper and Sloan	2008	500g	15s	Vickers
Nayif, Nakajima, Aksornmuang, Ikeda and Tagami	2008	50g	15s	Knoop
Brandt, de Moraes, Correr-Sobrinho, Sinhoreti and Consani	2008	50g	15s	Knoop

the fact that there is no significant difference between the methodologies and protocols tested.

The first null hypothesis affirms that there is no statistical difference when composite resin is submitted to knoop or Vickers microhardness using different testing protocols. The second hypothesis tested the possible correlation of elastic modulus of composite resin in microhardness tests. This article intends to discuss and question different microhardness protocols in order to avoid misinterpretations of results and trends by researchers that might occur.

Materials and Method

Nanohybrid composites resins (Grandio) samples were used in this study (*Table 2*) with different viscosities (medium n=270 and low n=270). The A2 composites were inserted in a single increment into a poly tetrafluoroethylene (PTFE) mould with 4 mm diameter and 2 mm high between two Mylar strips and flattened with a glass plate. Samples were photo cured using a LED device (Celalux, Voco, Cuxhaven, Germany, 800 mW/cm2) during 20s. After 24 h of storage at 37 °C with protection from light, samples were randomly divided into 36 groups according to the composite viscosity (medium or low), the methodology (VHN or KHN), load (50, 100 or 500g), and time (15, 30 or 45s) (*Figure 1*). The specimens were embedded in self-cured acrylic resin into PVC moulds in order to maintain the flattened surface. Two reading were made in the surface expose to the light of each sample leading

Table 2. *Description of materials used in the study.*

Trade mark	Viscosity	Composition	Batch #
Grandio	Medium	87 wt% of silicone dioxide and fine particles of glass BIS-GMA, TEGDMA	581271
Grandio Flow	Low	80 wt% inorganic filler 20 wt% BIS-GMA, TEGDMA, HEDMA	0846108

*According to manufacturer's instructions.

to 1080 indentations with Shimadzu HMV tester (Shimadzu, Kyoto, Japan). The Vickers (VHN) and Knoop (KHN) results were submitted to a three-way ANOVA with fixed factors the viscosity, load and time, and the post-hoc Tukey multiple comparison test at $\alpha = 0.05$.

Results

Significant differences were recorded ($p<0.001$) between groups in each tested methodologies for both viscosities (*Tables 3-4, Figures 2*). Among the Vickers (VHN) tested samples, the average recorded ranged from 164.94 (50g for 45s) to 210.33 (100g for 45s) for the medium viscosity composite and from 31.2 (100g for 30s) to 88 (500g for 45s) for a low viscosity resin.

Knoop microhardness (KHN) readings ranged from 128.92 (500g for 45s) to 184.26 (100g for 15s) for medium viscosity composite and from 23.6 (100g for 15s) to 73.4 (500g for 30s) for the low one.

Discussion

The aim of this study was to compare the methodologies of measurement of microhardness of composite resin using different protocols and load time application of composite resin with two viscosities. According Polydorou et al. 2007, two methodologies for analysis of microhardness of composite resin for its brittle and elastic behavior, Vickers and Knoop [10] are used. According to Neves et al. 2002, the methodology used to measure the Vickers hardness of the resin is based on a diamond pyramid with dihedral angle of 136 degrees which is compressed with an arbitrary force "F" to the surface of the material. Calculate the area "A" from the printed surface by measuring its diagonals that is applied to brittle materials. Already 7-1 possessed a Knoop diagonal ratio that is used to test the materials with an elastic behavior being able to shrink to smaller diagonal after removal of the load. The formulas used to make the measurements are based on a constant multiplied by an arbitrary force "F" on the diagonal to the square, and the formula for Vickers 1854.4 x F / D2 and the formula for Knoop 14.230 x F / D2 [6].

Significant differences were recorded between groups in both Vickers and Knoop methodologies and between the tested viscosities ($p<0.01$), therefore, the null hypotheses were rejected. Different loads and elapsed time during the assay can increase or decrease significantly microhardness results. Clinicians that commonly compare and choose materials according to the hardness must be aware.

However, in this study, it was observed that these formulas do not include all variables as time and load which directly influenced the outcome of the microhardness of composite resins by increasing or decreasing its value ($p<0.05$). Professionals in the field of dentistry using as reference the

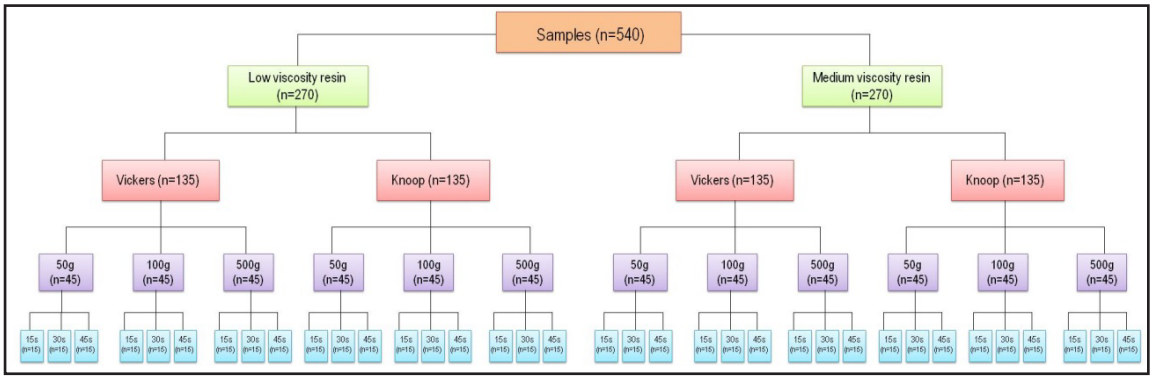

Figure 1. *Group distribution according to variables: viscosity, methodology, load and time.*

Table 3. *Average and statistical comparison of medium viscosity composite for methodology, load and time. (*Comparing averages in columns, different letters represent statistical difference when submitted to the Tukey Test (p < 0.05).*

	50g		100g		500g	
	Vickers	Knoop	Vickers	Knoop	Vickers	Knoop
15s	179.32^{bc}	168.13^{ab}	202.04^{ab}	184.25^{a}	181,58^{bc}	140.77^{bc}
30s	185.74^{abc}	149.87^{bc}	197.20^{ab}	179.13^{a}	193.76^{ab}	139.40^{c}
45s	164.94^{c}	180.33^{a}	210.33^{a}	148.09^{bc}	186.56^{abc}	128.92^{c}

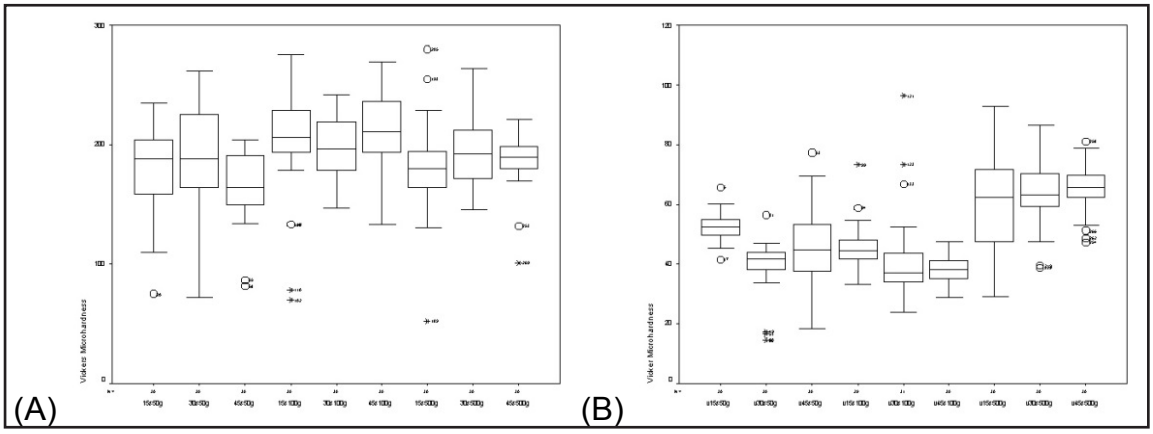

Figure 2. *Comparison of Vickers microhardness of medium (A) and low viscosities (B).*

Table 4. *Average and statistical comparison of low viscosity composite for methodology, load and time. (*Comparing averages in columns, different letters represent statistical difference when submitted to the Tukey Test (p < 0.05).*

	50g		100g		500g	
	Vickers	Knoop	Vickers	Knoop	Vickers	Knoop
15s	52.50^{bc}	41.63^{b}	46.14^{cd}	28.83^{c}	59.17^{ab}	63.03^{a}
30s	38.71^{d}	40.87^{b}	41.33^{d}	33.48^{c}	64.00^{a}	63.11^{a}
45s	45.82^{cd}	30.45^{c}	38.15^{d}	28.42^{c}	64.95^{a}	58.12^{a}

values of hardness, Vickers or Knoop, for selecting resin materials must be aware of the influence of these variables on the mechanical property toughness, in order to make future comparisons.

Based on the formulas was not expecting this behavior, as it was believed that they were able to compensate for variables Vickers and Knoop hardness of a composite resin did not differ regardless of its outcome chosen by the researcher [6-12] protocol. However, a change in load may reflect a significant result in the change of microhardness ($p < 0.05$). The elapsed time during loading is not covered by the formula, with the same significant results. Therefore research suggests that changes in load parameters and elapsed time are added in these formulas microhardness measurements.

Reviewing the literature, it is noted that there is no standardized protocol for measuring the hardness of the resins, which makes this comparison between the same dubious. There are all kinds of comparisons of results without taking into account the same measurement methodology [6,7,10-12]. This research reveals that, just based on the formula, the statement of a result of microhardness cannot be established. An association of variables such as the load and the elapsed time must be entered in the calculation of hardness in order to seek standardization of measurements which may define a comparison and in fact, to be reliable. To date, the clashes between the microhardness of resin should be performed with caution according to the protocol of each survey.

Conclusion

In a controlled *in vitro* experiment, the microhardness formulas mislead researchers to associate an improvement of diagonal measurements to higher load with a linear and proportional behavior. A new association of factors elastic

modulus and time shall be inserted. It is not recommended to clinicians compare both Vickers and Knoop microhardness results of composite materials without similar viscosities and protocols applied in the research.

References

1. Anusavice KJ. In: Phillips materiais dentários. 10.edn. Elsevier Ltda Rio de Janeiro. 2005; 41-43.

2. Sturdevant CM, Barton RE, Sockwell CL, Strickland WD (1985) The art and science of operative dentistry. 2nd ed., CV Mosby Co., St. Louis.

3. Garone NN, Carvalho RCR, Russo EMA, Sobral MAP, Luz MAAC (2003) Introdução à Dentística Restauradora. Editora Santos, São Paulo.

4. Tyas MJ, Jones DW, Rizkalla AS. The evaluation of resin composite consistency. *Dental Materials*. 1998; **14**: 424-428.

5. Gomes JC. Análise in vitro da microinfiltração marginal em cavidades de classe II restauradas com resina composta de alta densidade, utilizando uma resina "flow" como "liner" em esmalte e em dentina. *Jornal Brasileiro de Dentística & Estética*. 2002; **1**: 269-301.

6. Neves AD, Discacciati JA, Orêfice RL, Jansen WC. Correlation between degree of conversion, microhardness and inorganic content in composites. *Pesquisa Odontológica Brasileira*. 2002; **16**: 349-354.

7. Rode KM, Kawano Y, Turbino ML. Evaluation of curing light distance on resin composite microhardness and polymerization. *Operative Dentistry*. 2007; **32**: 571-578.

8. Brandt WC, de Moraes RR, Correr-Sobrinho L, Sinhoreti MA, Consani S. Effect of different photo-activation methods on push out force, hardness and cross-link density of resin composite restorations. *Dental Materials*. 2008; **24**: 846-850.

9. Mota EG, Oshima HM, Burnett LH Jr, Pires LA, Rosa RS. Evaluation of diametral tensile strength and Knoop microhardness of five nanofilled composites in dentin and enamel shades. *Stomatologija*. 2006; **8**: 67-69.

10. 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*. 2007; **23**: 153-8.

11. Aguiar FH, Braceiro A, Lima DA, Ambrosano GM, Lovadino JR. Effect of light curing modes and light curing time on the microhardness of a hybrid composite resin. *Journal of Contemporary Dental Practice*. 2007; **8**: 1-8.

12. Voltarelli FR, dos Santos-Daroz CB, Alves MC, Peris AR, Marchi GM. Effect of different light-curing devices and aging procedures on composite knoop microhardness. *Brazilian Oral Research*. 2009; **23**: 473-479.

13. Price RB, Fahey J, Felix CM. Knoop microhardness mapping used to compare the efficacy of LED, QTH and PAC curing lights. *Operative Dentistry*. 2010; **35**: 58-68.

14. Correa MB, Henn S, Marimon JL, Rodrigues SA Jr, Demarco FF. Factors influencing the microhardness of a microhybrid composite. *General Dentistry*. 2010; **58**: 94-98.

15. Fleming GJ, Awan M, Cooper PR, Sloan AJ. The potential of a resin-composite to be cured to a 4mm depth. *Dental Materials*. 2008; 24: 522-529.

16. Nayif MM, Nakajima M, Aksornmuang J, Ikeda M, Tagami J. Effect of adhesion to cavity walls on the mechanical properties of resin composites. *Dental Materials*. 2008; **24**: 83-89

17. 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*. 2007; **49**: 19-24.

18. Yazici AR, Kugel G, Gül G. The Knoop hardness of a composite resin polymerized with different curing lights and different modes. *Journal of Contemporary Dental Practice*. 2007; **8**: 1-8.

19. Tango RN, Sinhoreti MA, Correr AB, Correr-Sobrinho L, Consani RL. Effect of veneering materials and curing methods on resin cement knoop hardness. *Brazilian Dental Journal*. 2007; **18**: 235-239.

20. Catelan A, Santo MR, Menegazzo LM, Moraes JC, dos Santos PH. Effect of light curing modes on mechanical properties of direct and indirect composites. *Acta odontologica Scandinavica*. 2013; **71**: 697-702.

21. Erdemir U, Sancakli HS, Yildiz E. The effect of one-step and multi-step polishing systems on the surface roughness and microhardness of novel resin composites. *European Journal of Dentistry*. 2012; **6**: 198-205.

22. Sabatini C. Comparative study of surface microhardness of methacrylate-based composite resins polymerized with light-emitting diodes and halogen. *European Journal of Dentistry*. 2013; **7**: 327-35.