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Effect of Indentation Load and Time on Knoop and Vickers Microhardness Tests for Enamel and Dentin

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The aim of this study was to determine the effect of variations in indentation load and time on the Knoop and Vickers hardness numbers (KHN and VHN) for enamel and dentin. Twenty molar teeth were divided into twenty enamel and twenty dentin specimens. Each specimen was tested using a Knoop or Vickers microhardness tester at different loads and times. The difference in hardness between the groups was analyzed with two-way ANOVA followed by a Tukey test. The results revealed that a difference of indentation time did not influence the microhardness number of enamel and dentin. The KHN values of enamel and the VHN values of dentin were affected by variation of test loads. Therefore, the tooth hardness number for different loads may not be acceptable for comparison.

Keywords: *dentin, enamel, Knoop hardness, Vickers hardness*

1. Introduction

Microhardness tests are commonly used to study the physical properties of materials, and they are widely used to measure the hardness of teeth[1-3]. This method is easy, quick, and requires only a tiny area of specimen surface for testing. Using this technique, the specimen surfaces were impressed with a diamond indenter (a Knoop or a Vickers) at a certain load for a certain period of time. After load removal, diagonals of the indentation were measured with an optical microscope. The hardness number was defined by the ratio between the indentation load and the area of the residual impression, which depended on the indenter shape. Then the hardness of materials was calculated using these equations:

$$HK = 14230\ (F/d^2) \qquad \text{for Knoop microhardness}$$

or

$$HV = 1854\ (F/d^2) \qquad \text{for Vickers microhardness}$$

The constant value of each equation was calculated from the specific geometry of the indenter; F was the indentation load (g), and d was the diagonal of the indentation (µm).

The indentation load for the microhardness test can be performed using 1 to 1,000 g, and with various loading times. There is no agreement on the specifications of the conditions for the test method of a tooth. An examination of the literature reveals that previous studies have used different indenters and various loads and times to investigate the hardness of enamel and dentin[4-8].

From the HK and HV equations, the microhardness value should be constant when loads are varied, because the indentation size increases with an increase in the load. However, studies of microhardness results on a wide range of loads have shown that results are not constant at very low loads[9]. This characteristic can be attributed to elastic recovery or the viscoelastic nature[10], the grain size effect[11], indentation cracks, surface texture, or diagonal measurement errors[12].

Enamel and dentin have specific microstructures; thus their hardness may depend upon indentation loads or times. The lack of this information makes it difficult to compare the results of prior studies which had the same purpose, but which used different indentation loads and times. The aim of this study was to evaluate the effect of indentation loads and times on Knoop and Vickers microhardness tests for human enamel and dentin.

2. Materials and Methods

Twenty enamel specimens and twenty dentin specimens were prepared from twenty human third molar teeth. Each tooth was cut longitudinally, to separate the buccal and lingual halves, using a diamond blade saw (IsoMet 4000, Buehler, USA). Each specimen was then embedded in a resin block. The middle region of the buccal half was ground to provide a flat surface approximately $2 \times 2\ mm^2$ using silicon carbide paper with grit sizes 320, 600 and 1200, and polished using 1 µm diamond suspension. Enamel from the lingual half was removed to expose a dentin surface approximately $3 \times 3\ mm^2$, to serve as a dentin specimen. Ten enamel specimens and ten dentin specimens were used for a Vickers microhardness test; the other ten enamel and ten dentin specimens were used for a Knoop microhardness test. Using a microhardness tester (Micromet II, Buehler, USA), each enamel specimen was impressed with loads of 100, 200 and 300 g for 10, 20 and 30 seconds, and each dentin specimen with indentation loads of 10, 25 and 50 g for 10, 20 and 30 seconds using a Knoop or a Vickers indenter. The minimum spacing of indents was 2.5 times the indent diagonal, as demonstrated in Figure 1. Each test condition with the same load and time was conducted three times; thus there were 27 indentations on each specimen surface obtained from different test loads and times. An average of three readings for each test condition was recorded as the KHN or VHN value of a specimen. Data of each experimental condition from ten specimens were averaged, and differences in KHN or VHN values were compared using two-way ANOVA followed by a Tukey test.

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3. Results

The results of the Knoop and Vickers hardness tests for enamel and dentin at different test loads and times are shown in Figures 2-5. For enamel, the average KHN values are in the range of 315.7-354.1 kg.mm^{-2}, while VHN values are in the range of 316.0-328.4 kg.mm^{-2}. For dentin, under the same testing conditions, the data shows without ambiguity that KHN values, which varied from 55.2 to 61.6 kg.mm^{-2}, are higher than VHN values, which varied from 45.7 to 54.9 kg.mm^{-2}.

In Table 1, two-way analysis of variance revealed statistically significant differences at different indentation loads in KHN of enamel and VHN of dentin. Multiple comparison with a Tukey test revealed that for various loading times the average KHN value of enamel at a load of 100 g differed from a load of 300 g. Also the average VHN value of dentin at a load of 10 g differed from loads of 25 and 50 g. However, there were no statistically significant differences of VHN values in enamel and KHN values in dentin at different loads. Different indentation times did not affect these test results. The interaction between different loads and different times was tested, but showed no statistical significance.

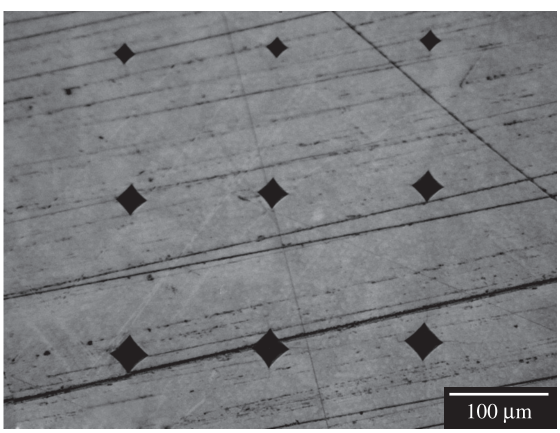

Figure 1. Group of Vickers indentations on enamel at different loads and times.

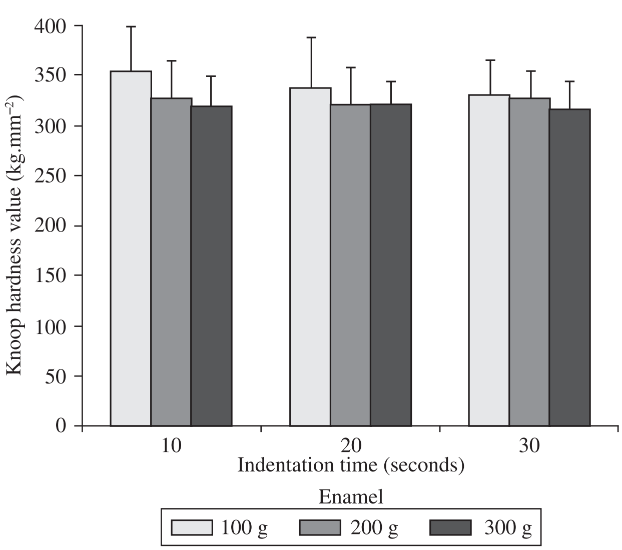

Figure 2. Average KHN values (SD) of enamel at different indentation loads and times.

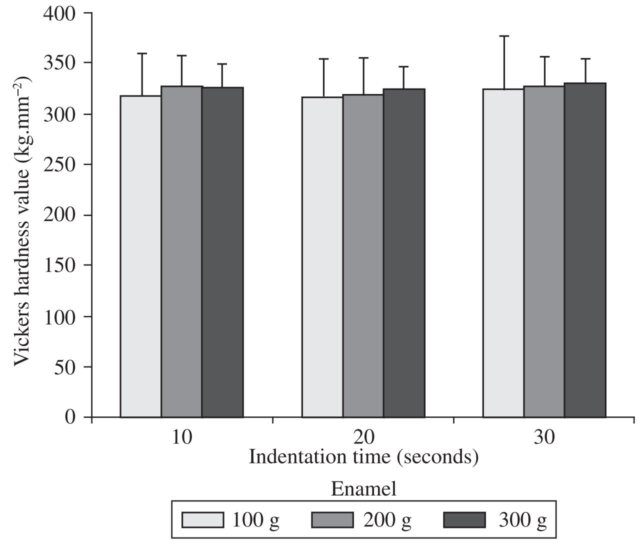

Figure 3. Average VHN values (SD) of enamel at different indentation loads and times.

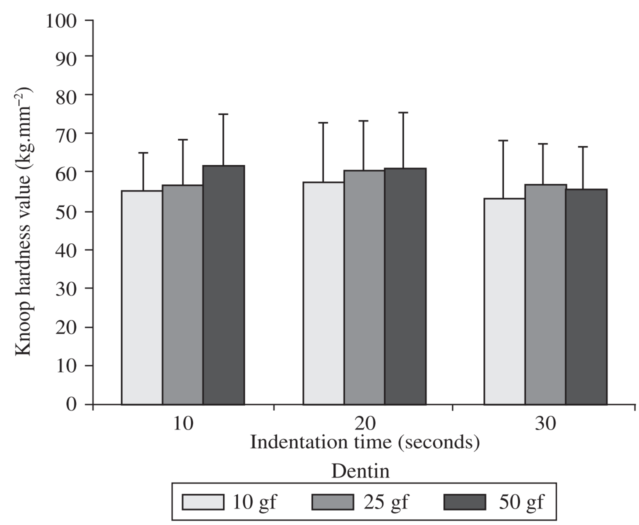

Figure 4. Average KHN values (SD) of dentin at different indentation loads and times.

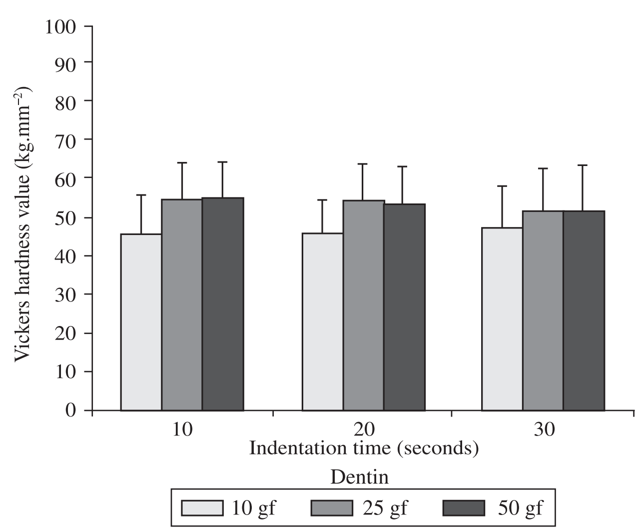

Figure 5. Average VHN values (SD) of dentin at different indentation loads and times.

Table 1. Results of two-way ANOVA.

Hardness type	F value		
	Load	Time	Load × time
Enamel			
KHN	**6.521***	0.757 ns	0.695 ns
VHN	2.834 ns	0.738 ns	0.685 ns
Dentin			
KHN	2.326 ns	2.856 ns	0.409 ns
VHN	**14.044***	0.725 ns	0.614 ns

*Significance at $P < 0.05$; ns: not significant.

4. Discussion

There is no standard condition for enamel and dentin microhardness testing; therefore, selection of testing conditions depended on the researcher's decision. Numerous previous microhardness studies reported results of both KHN and VHN at different indentation loads and times[4-8,13-15]. There are many reasons to perform tests at different conditions. A high load is chosen for the reason that it produces a large impression, and it is thus easy to measure the indentation diagonal. However, a high load applied on a soft surface causes an oversize impression, where the diagonals are longer than the micrometer scale fitted to the eyepiece of the tester. Therefore in a pre-post experimental study of, for instance, enamel erosion, it is necessary to apply a small load for a comparison between the baseline surface and the eroded surface for the same indentation load. Gutiérrez-Salazar and Reyes-Gasga[16] proposed that in tooth hardness studies the Vickers indenter is more useful than the Knoop because a square shape must always be conserved, and because the indentation produced on a non-flat surface, or by the difference in hardness of enamel and dentin, is easily detected. Meredith et al.[17] reported that the Knoop has been the most popular method. Knoop indentation is longer and shallower than Vickers indentation, so a load impression can be applied to brittle materials without cracking. Plus, the longer diagonal is easier to read than the short diagonal of the Vickers. However in this study, the advantage of the Knoop's longer diagonal was offset by the difficulty in deciding where the tapered tip ends on the surface of the dentin.

Without considering variations of indentation loads and time, the results obtained for the KHN and VHN values of enamel and dentin in this study are in accordance with previously published values: for example, the hardness of enamel has been reported in the range of 314 to 361 KHN[18] or 322 to 353 VHN[6]. For dentin, the hardness has been reported in the range of 52 to 64 KHN[14,19] or 46 to 53 VHN[15]. The standard deviation of hardness in this study showed a broad variation, similar to previous reports[16]. This broad variation of hardness values can be produced by factors such as specimen preparation, diagonal length reading error[16], variation in chemical composition[20], age, and location in the tooth[21].

In this study, the KHN and VHN values of enamel are in approximately the same range, while dentin obtained KHN values higher than VHN values for every variation of indentation load and time. Many researchers have tried to explore the relationship between these two hardness parameters. Mukhopadhyay et al.[22] suggested that KHN and VHN values could be correlated by a calibration factor; but Gong et al.[23] indicated that the relationship may be more complex than that predicted for various materials by one relationship equation. However, Chicot et al.[24] indicated that KHN values are lower than VHN values for hard materials which exhibit high hardness. For soft materials which have low hardness, KHN values are greater than VHN values. Therefore the difference between KHN and VHN for each material should be obtained from measurements using a microhardness tester.

The tester can select a load ranging from 1 to 1,000 g. Test loads of 100, 200 and 300 g for enamel and 10, 25 and 50 g for dentin were chosen for comparison purposes in this study because they have been used in a number of previous studies. A load of 300 g for enamel and 50 g for dentin produced a Knoop indentation diagonal of approximately 100 µm, while the 400× magnification of the attached optical microscope used in this study can measure a maximum length of about 300 µm. A higher load may be impractical for a softer surface in the pre-post experiment because, after treatment, it produces a larger impression than the optical microscope can measure. The lowest loads, 100 g for enamel and 10 g for dentin, were chosen for this study because they created Vickers diagonals longer than 20 µm, which was recommended to prevent errors in optical measurment[25].

Two-way ANOVA (Table 1) showed that the KHN values of enamel and the VHN values of dentin depended on the indentation load. Nevertheless, the variation loads did not statistically have any effect on VHN values of enamel and KHN values of dentin. The phenomenon that microhardness values depend on the indentation load – the indentation size effect (ISE) – is well-known, and has been observed in many materials. Sangwal[26] indicated that there are two types of ISE: normal ISE, which usually involves a decrease in microhardness with increasing indentation load; and reverse ISE, where microhardness increases with increasing indentation load. The ISE that occurred in this study was either the KHN of enamel showing normal ISE, or the VHN of dentin showing reverse ISE. There are two groups of factors that may contribute to ISE[10,26,27]. The first is the hardness measurement accuracy, and the influence of indenter geometry on hardness. Uncertainties in the measurement of small indentation areas, particularly when pile-up or sink-in effects are present, can lead to over- or underestimation of the indentation area. The second factor contributing to the ISE effect is caused by the properties of the materials. Shahdad et al.[28] discussed the ISE effect associated with elastic recovery after indenter removal, and elastic-plastic deformation under the indenter, particularly at low test loads, and that there can be further complications if it occurs in brittle materials. They found that none of the ISE models can be accepted as the best to describe the ISE in different types of materials[10]. Enamel is a brittle material and has a hierarchical structure. Dentin is hard and has a cellular component. Therefore, it is not appropriate to compare microhardness values which are obtained by different indentation loads. The analysis of possible causes of the ISE phenomenon would be interesting to investigate in a further study.

The difference of loading times (10, 20 and 30 seconds) was not significant for either enamel or dentin tested at the same test load. This suggests that an indentation time of 10 seconds is sufficient for a permanent indentation on the tooth surface to take place.

5. Conclusions

The results of this study showed that the difference of indentation times was not influential on KHN and VHN values of enamel and dentin for the same indentation loads. The KHN values of enamel and the VHN values of dentin were, however, affected by variation of indentation loads.

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