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CORRELATION OF THE HUMAN DENTINE HARDNESS AND ELASTIC PROPERTIES

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Abstract: *The paper presents a study performed on human dentine, in order to correlate the micro hardness values measured with elastic evolution of the indentation and mechanical behavior of the ceramic biocomposite. Statistical correlations were made between the indentations elastic recovery and the morphology and normalization formulae was used to establish a correct value for the Vickers micro hardness index .*
Keywords: *biocomposite, dentine, micro hardness, elastic recovery*

1. INTRODUCTION

Knowing the real macro and micro properties of the human tooth tissues, is very important for the choice of compatible building materials, with a behavior similar with the natural biocomposites. Of a great importance is the behavior at dynamic, cyclic loads or repetitive temperature variations. The actual elastic modulus and hardness values can be of a great help in understanding how the man-made materials can absorb and dissipate the stresses, both locally and in the tooth block.
This study aims to determine the human dentine hardness using the micro hardness Vickers index and to analyze the indentation force correlated with the indentation time, and to establish an effective method to take into account the elastic recovery phenomenon, when stating the Vickers index. This value is to be further linked with mechanical and chemical properties of the dentine.

2. THE ANALYSIS METHOD

Using an HV-1000 apparatus, and several forces with corresponding application durations (10gf, 25gf, 50gf for 10s,15s, 30s) [1], for each dentine sample incorporated in polymer support (figure 1) were made 10 indentations, at appropriate distance (larger than 2.5 max. diagonal) [1, 2]. The indentations were check for symmetry and filtered. Figure 1 presents the samples and indentations, figure 2 presents detailed view for sampled indentations for various load at the same measurement time. Figure 3 shows the distribution of the HV values on the measured length L, at 25gf/10s.

Figure 1: The dentine samples and a capture of the indentations

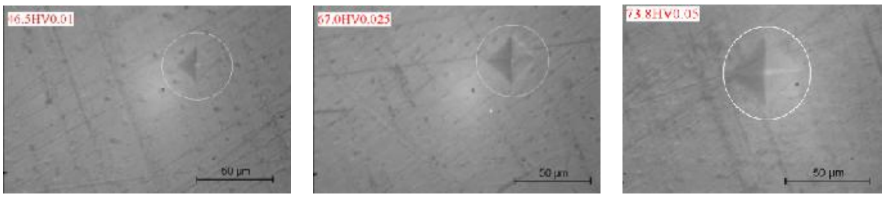

Figure 2: Indentations in human dentine for: 10gf/10s, 25gf/10s, 50gf/10s.

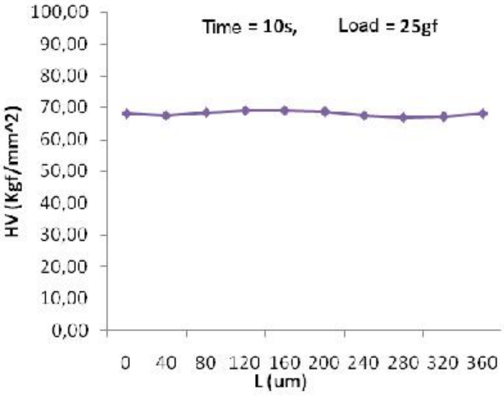

Figure 3: HV values on the measured length L, at 25gf/10s

For each sample, the arithmetic mean, standard deviation, asymmetry quotients were calculated (figure 4). The One Way ANOVA test [3] confirmed that there are not significant differences among hardness values for the ten indentations per sample, each set being obtained for different force and time values (F<Fcr, p>0,05).

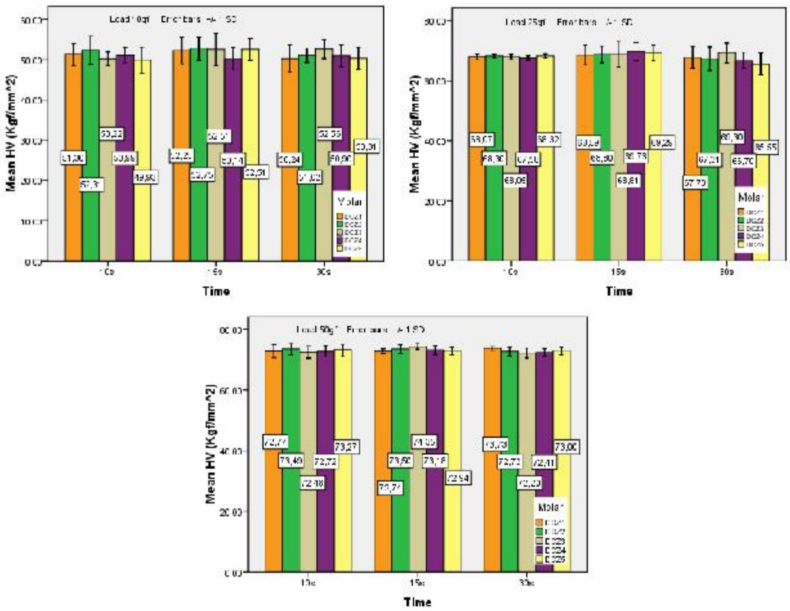

Figure 4: Comparison of the human dentine HV values, for 10, 25 and 50 gf at different load durations.

The result of this preliminary analysis stated that, for a given duration and load, the hardens values obtained for each sample can be associated in simple data sets of 50 values, fitted for further statistical analysis [4, 5]. The One Way ANOVA applied for these data sets showed that, for a given load, there are no significant differences among the hardness values (F<Fcr, p>0,05), depending of the indentation load of, respectively, 10s, 15s and 30s (figure 5).

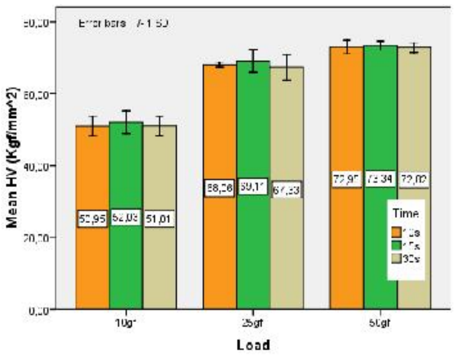

Figure 5: Comparison of the dentine HV values at a given load and different durations.

The unifactorial analysis One Way ANOVA applied to the 50 component data sets [5] revealed that, for a given indentation duration, there are significant differences amongst the HV values (F>Fcr, p<0,05), depending on the indentation loads of, respectively, 10gf, 25gf and 50gf. This conclusion is sustained also by Post-Hoc Multiple Comparisons with a probability of p< 0,001 in all cases, the largest value registered corresponding to the largest load of 50gf.

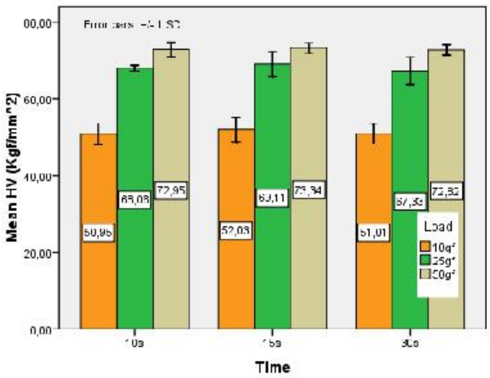

Figure 5: Comparison of the dentine HV values at a given duration and different loads.

The data were analyzed with Two Way ANOVA [6], with several hypotheses regarding: the global effect of the factors involved in hardness determination (load and duration), considered altogether, indistinct; principal effects, for each analysis factor and cumulative effect of the independent variables (force and load) on the dependent one (HV) (table 1).

Table 1: Tests of Between-Subjects Effects

Dependent Variable:HV (Kgf/mm^2)

Source	Type III Sum of Squares	df	Mean Square	F	Sig.	Partial Eta Squared
Corrected Model	39041,190[a]	8	4880,149	799,938	,000	,936
Intercept	1853378,493	1	1853378,493	303799,753	,000	,999
Load	38916,232	2	19458,116	3189,511	,000	,935
Time	100,866	2	50,433	8,267	,000	,036
Load * Time	24,093	4	6,023	,987	,414	,009
Error	2690,390	441	6,101			
Total	1895110,074	450				
Corrected Total	41731,581	449				

a. R Squared = ,936 (Adjusted R Squared = ,934)

Table 1 reveals that, although the global effect is significant, (F = 799,93 and p < 0.001), a detailed analysis shows that the dentine hardness values is separately intensely influenced by the load (F = 3189,5 cu p < 0,001), with an intensity of η2 = 0,935, while the indentation duration has a smaller influence (F = 8,26 cu p < 0,001) with an intensity of η2 = 0,036; the cumulative influence of the two factors is insignificant (F = 0,987 cu p = 0,414).

3. HUMAN DENTINE ELASTIC BEHAVIOUR

3.1. The elastic recovery index for the indentation depth correction

After the hardness test, important modification in the imprint geometry occur, if the material limits are not over passed. In order to take into account the elastic recovery of the biocomposite, when a micro hardness value is obtained via indentation, an supplementary index is needed – the elastic recovery index I_{er} (1), to reflect the imprint depth modification after the indentation [7].

$$I_{er} = \frac{H_t - H_{meas}}{H_t} \cdot 100\% \quad (1)$$

In formula 1, a theoretical depth is used H_t. Assuming that the diagonals remain unchanged and the recovery takes place mostly on the normal direction of the tested surface (up to 50%), the theoretical depth useful for a valid normalized quotient is:

$$H_t = \frac{d_1 + d_2}{2} \cdot ctg\varphi, \quad (2)$$

where $d_{1,2}$ are the two diagonals of the imprint and φ is the half of the indenters pyramid angle of 74,05°.
The measured depth *Hmeas* is the largest of the measured imprint's depth, in absolute value. Table 1 reveals that the elastic recovery is more important at lower indentation loads.

Table 2: Measured and theoretical indentations depths

Load		N	Minimum	Maximum	Mean	Std. Deviation
10gf	Ht x 10-2 (mm)	150	,5079	,5775	,543818	,0153250
	Hmeas x 10-2 (mm)	150	,3100	,3800	,348617	,0194595
	Ier (%)	150	27,05	45,66	35,8287	4,24592
25gf	Ht x 10-2 (mm)	150	,7020	,7877	,745741	,0156773
	Hmeas x 10-2 (mm)	150	,4700	,5400	,508229	,0165789
	Ier (%)	150	25,02	38,90	31,8177	2,68540
50gf	Ht x 10-2 (mm)	150	,9942	1,0514	1,018389	,0108114
	Hmeas x 10-2 (mm)	150	,7769	,8400	,809393	,0123180
	Ier (%)	150	17,15	24,48	20,5147	1,40194

3.2. The hardness value normalization

As a consequence of the nonlinearity between the hardness and the elastic recovery, a 4th grade inverse dependence is fit to describe this relationship [8]:

$$HV_n = \frac{1,854 \cdot P}{d^2} \cdot \sqrt[4]{I_{er} \cdot C}, \quad (3)$$

where *C* is a constant with a specified value of 2.5 [9], while *P* is the indentation load and d is the arithmetic mean of the diagonals if the imprinted pyramid. The hardness values, calculated before and after normalization, and the corresponding elastic recovery index are presented in table 3.

Table 3: HV values before and after normalization

Load		N	Minimum	Maximum	Mean	Std. Deviation
10gf	HV (Kgf/mm^2)	150	45,41	58,70	51,3288	2,89651
	HVn (Kgf/mm^2)	150	44,22	56,83	49,8226	2,33536
	Ier (%)	150	27,05	45,66	35,8287	4,24592
25gf	HV (Kgf/mm^2)	150	61,02	76,82	68,1664	2,85082
	HVn (Kgf/mm^2)	150	58,66	71,84	64,3014	2,24951
	Ier (%)	150	25,02	38,90	31,8177	2,68540
50gf	HV (Kgf/mm^2)	150	68,50	76,60	73,0342	1,54209
	HVn (Kgf/mm^2)	150	58,62	65,20	61,7673	1,19126
	Ier (%)	150	17,15	24,48	20,5147	1,40194

Table 4: Paired Samples Test

HV (Kgf/mm^2) - HVn (Kgf/mm^2)			Load		
			10gf	25gf	50gf
Paired Differences		Mean	1,50620	3,86501	11,26686
		Std. Deviation	1,54748	1,45777	1,19599
		Std. Error Mean	,12635	,11903	,09765
	95% CI of the Diff.	Lower	1,25653	3,62981	11,07390
		Upper	1,75588	4,10021	11,45982
t			-6,486	7,335	11,921
df			149	149	149
	p (Sig.) 2-tailed		,000	,000	,000

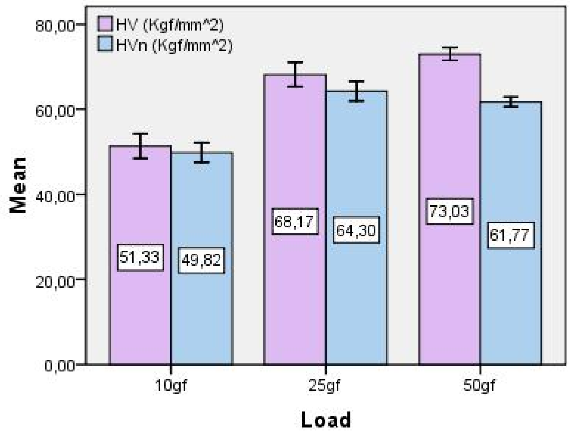

Figure 6: HV and HVn comparison

With Paired Samples Test (table 4), it becomes obvious that the hardness values measured after indentation HV (Kgf/mm^2) and the normalized ones HVn (Kgf/mm^2), are significant different with p (Sig.)< 0,001.

4 CONCLUSIONS

This study aimed to validate a simple method to take into account the complexity of the human dentine behavior and to include the elastic recovery in the hardness measurements. Because under the load the material is more deformed than after the indenter release, even the immediate measurements of the imprint can induce errors in hardness values. These errors can be avoided using a normalization quotient that takes into account the depth variation and the nonlinear 4th degree relationship between the hardness and the elastic recovery.

A thorough measurement sequence was established using a micro hardness tester, samples of dentine and corresponding indentation under specific standard loads and durations. After the statistical verification of the coherence of the measurements, cohorts of data were established for further analysis, to determine the most influent factor on the hardness (the load).

The elastic recovery takes place mostly on the normal of the indentation, while the transversal dimensions remain, practically, unchanged. The depth measurements immediately after the indentation and, respectively, after 12 hours showed depth recoveries up to 20-30%, as the indentation load grows, confirming that the dentine has better elastic properties at lower stresses and its capacity to reformulate it's structures decreases as local stresses increase.

The normalization decreases the HV values, less for the values obtained with 10gf loads and more for bigger loads, both being dragged near a mean value, which is to be taken into account for dentine mechanical characterization (table 5).

Table 5: Measured and normalized HV behavior for various indentation loads

Load		Mean	Std. Deviation
10gf	HV (Kgf/mm^2)	51,3288	2,89651
	HVn1 (Kgf/mm^2)	**49,8226**	2,33536
	Ier (%)	35,8287	4,24592
25gf	HV (Kgf/mm^2)	68,1664	2,85082
	HVn1 (Kgf/mm^2)	**64,3014**	2,24951
	Ier (%)	31,8177	2,68540
50gf	HV (Kgf/mm^2)	73,0342	1,54209
	HVn1 (Kgf/mm^2)	**61,7673**	1,19126
	Ier (%)	20,5147	1,40194

The statistical tests applied to the measured and normalized values confirmed the validity of the normalization procedure, with a high rate of confidence.
Further research can link the hardness value with other, non-mechanical properties of the dentine.

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