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On the possibility of estimating the fracture toughness of enamel

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ABSTRACT

Objectives. There are many works that have attempted to estimate the fracture toughness of enamel by indentation techniques using equations whose success in determining the actual value of fracture toughness, rely on a particular three-dimensional pattern consisting of cracks growing from the edges of the indentation. Recently, an alternative methodology based on an energetic approach has been developed to estimate the fracture toughness of coatings by depth sensing indentation that is not less affected by the cracks pattern generated. In this work, the energetic approach to indentation fracture toughness of bovine enamel is presented and compared with those toughness values obtained using the traditional expressions reported in the literature.

Methods. Indentation tests were carried out using a diamond Berkovich indenter onto the enamel surface of eight incisors from bovines of two years old. A continuous stiffness measurement methodology was used with a frequency of 45 Hz and displacement amplitude of 2 nm up to a maximum penetration depth of 2000 nm.

Results. The results showed that some modifications in the energetic methodology should be performed in order to apply it successfully.

Significance. The fracture toughness values obtained using the traditional equation and applying the energetic methodology, were significantly different, although the values were within the range obtained by other authors.

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1. Introduction

Enamel is the hardest and stiffest tissue of mammals. Its microstructure consists of rods encapsulated by thin protein rich sheaths that are arranged parallel in a direction perpendicular to the dentino-enamel junction (DEJ) from dentin to the outer enamel surface.

The enamel microstructure of all mammals appears to be very similar on a histochemical and anatomic basis [1–5].

Numerous methods have been employed to experimentally measure the fracture toughness (K_C) of the enamel. The

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determination of K_C by indentation techniques is based on measuring the size of cracks induced in a material during indentation [6]. Several expressions are available to determine K_C by this technique, depending on the indenter geometry and crack morphology [7–10].

In 1987, Laugier [10] determined that:

$$K_C = x_V \left(\frac{a}{l}\right)^{1/2} \left(\frac{E}{H}\right)^{2/3} \frac{P}{c^{3/2}} \tag{1}$$

where a is the half-diagonal of the indentation impression, l is the crack length measured from the indentation imprint edge, and c is the crack length measured from the center of the indentation imprint. Additionally, Laugier [10] showed that the radial and half-penny models make similar predictions when $x_v = 0.015$.

The Eq. (1) was developed for ceramic materials and for the symmetrical Vickers indentations. Some efforts have been made to obtain similar equation but properly modified for Berkovich indentations. Ouchterlony [11] investigated the nature of the radial cracking and determined a modification factor for stress intensity factor to account the number of radial cracks, n, formed.

$$k_1 = \sqrt{\frac{n/2}{1 + (n/2\pi)\sin(2\pi/n)}} \tag{2}$$

The ratio of k_1 values for $n = 4$ (Vickers indenter) and $n = 3$ (Berkovich indenter) is 1.073. Introducing this ratio in Eq. (1), a modified Laugier model adapted to Berkovich indentations can be obtained, according to Eq. (3).

$$K_C = 1.073 \cdot x_V \left(\frac{a}{l}\right)^{1/2} \left(\frac{E}{H}\right)^{2/3} \frac{P}{c^{3/2}} \tag{3}$$

However, these equations provide an actual value of the fracture toughness by indentation only if a particular pattern of cracks running from the indentation imprint vertices is obtained.

This work has been developed to answer the question about what happens if cracks generated by indentation do not show that characteristic pattern. Concerning this point, there could be an alternative methodology based on the energy released during cracking process, as a measurement of the fracture toughness [12]. This energetic method calculates the fracture toughness comparing the area limiting by the load-displacement curve with crack generation and the hypothetical one if cracking does not occur [12].

For a thin film, Li et al. [12] determined the critical stress-intensity factor assuming Mode I:

$$K_{IC} = \left[\left(\frac{E}{(1-\nu^2)2\pi C_R}\right) \cdot \left(\frac{\Delta U}{t}\right)\right]^{1/2} \tag{4}$$

where E is the elastic modulus and ν is the Poisson's ratio, K_{IC} is the indentation fracture toughness of the material, ΔU is a measure of the fracture energy, t is the film thickness and $2\pi C_R$ is the crack length. The product of $2\pi C_R$ and t gives the cracked area, A_{crack}, therefore, Eq. (4) can be written as:

$$K_{IC} = \left[\left(\frac{E}{(1-\nu^2)}\right) \cdot \left(\frac{\Delta U}{A_{crack}}\right)\right]^{1/2} \tag{5}$$

Eq. (5) allows obtaining fracture toughness values from indentation tests. Under this testing method, the strain energy, ΔU, can be obtained from the area between the hypothetical loading curve if no cracking exists and the experimental one. This equation can be used even when a particular cracks pattern is not obtained, although the critical point of Eq. (5) is to determine again the cracked area.

Hereafter, Eq. (3) is referred in this paper as the traditional equation to distinguish the equation using the energetic methodology, Eq. (5).

There is some scatter in the literature about the actual fracture toughness of enamel [13]. Hassan et al. [14] have reported values of human tooth enamel, using a Vickers indenter, in the range of 0.7–1.37 MPa m$^{1/2}$. Xu et al. [15] reported fracture toughness values of 0.84 MPa m$^{1/2}$ for labial human enamel also using a Vickers indenter in the same range of those determined by Padmanabhan et al. [16]. Bajaj and Arola [17] reported fracture toughness values for human enamel that ranged from 1.79 MPa m$^{1/2}$ to 2.37 MPa m$^{1/2}$ obtained from R-curve analysis, and Baldassarri et al. [18] obtained values of 0.5 MPa m$^{1/2}$ and 1.3 MPa m$^{1/2}$ for transversal and midsagital enamel orientation, respectively, using a Vickers indenter on rat tooth. There are various reasons that may cause the large variation in reported fracture toughness values. The biological nature of the enamel suggests that the compositional variations among specimens could impact to the toughness values [19,20]. Additionally, the microstructure orientation of enamel could also contribute to this variability [21].

Therefore, the aim of this study was to compare the fracture toughness values obtained from the energetic methodology using depth sensing indentation technique with those obtained applying the traditional equation based in a specific pattern of cracks.

2. Materials and methods

2.1. Preparation of specimens

Eight incisors were extracted from bovines of two years old. Teeth were cleaned and stored in artificial saliva (Table 1) prior to the tests. The labial surfaces of the specimens were polished

Table 1 – The composition of artificial saliva.

Composition	g/100 g of solution
Sodium carboxymethyl cellulose	1.000
Sorbitol	3.000
Sodium chloride	0.084
Potassium phosphate	0.120
Calcium chloride, dihydrate	0.015
Magnesium chloride, hexahydrate	0.005
Dibasic sodium diphosphate	0.034
Purified water	bal

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