J Mater Sci: Mater Med (2007) 18:139–142
DOI 10.1007/s10856-006-0672-y

In vitro microhardness of glass ionomer cements

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Received: 11 July 2005 / Accepted: 14 November 2005

Abstract This study evaluated the surface microhardness of four glass ionomer cements and a composite resin (Fuji IX, Ketac Molar, Vidrion R, Vitromolar and Z 250). Ten specimens of each glass ionomer cement with 8.0 mm diameter and 5.0 mm high dimensions were made and Vicker's microhardness measurements were taken at 1 day and 1 week after initial setting reaction. The results were analyzed using Student's T test and Tukey test ($p < 0.05$) and demonstrated that the values of microhardness increased after 1 week, with the exception of Fuji IX. Resin composite Z250 presented the greatest values for microhardness.

1 Introduction

In spite of the development of extensive preventive and curative treatment techniques available to dentists, dental caries is an increasing and largely untreated problem in many developing countries, where the relief of severe pain is usually achieved by tooth extraction [1–3].

The introduction of the Atraumatic Restorative Treatment (ART) approach represents a simple, low cost and patient acceptable alternative to the conservative management of dental caries. The ART technique involves the removal of carious tissue with hand instruments and restoration of the cavity and sealing of any adjacent enamel fissures with a conventional self-hardening glass ionomer cement [4–7].

Glass ionomer cements were developed to combine the advantages of biological and adhesive properties of dental silicate and zinc polycarboxylate cements [8]. These materials present certain properties, such as chemical bonding to enamel and dentine, release of fluoride and low coefficient of thermal expansion similar to teeth. They are, however, susceptible to fracture and exhibit low wear resistance. These disadvantages limited their indication for areas subject to strong masticatory stress [9].

Due to such deficiencies in glass ionomers, efforts have been made to improve their mechanical properties by incorporating metal or resin based glass ionomer cements. Recently, new fast-set highly viscous glass-ionomer cements were introduced. This fast setting reaction may result in superior mechanical properties and good wear resistance [9].

As microhardness testing can be performed to evaluate the setting reaction of the glass ionomer cements [10], the aim of this study was to evaluate the microhardness of two highly viscous glass ionomer cements (Fuji IX and Ketac Molar) and two conventional glass ionomer cements (Vidrion R and Vitromolar) used in the ART approach. Composite resin was used as a control group.

2 Materials and methods

Materials used in this study are shown in Table 1. For microhardness testing, Teflon moulds were used with internal circle perforation dimensions of 8.0 mm in diameter and 5.0 mm high (Fig. 1). Ten specimens were made for each material.

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Table 1 Manufacturer, presentation form and color of the materials used in the study

Trade mark	Manufacturer	Presentation	Color
Z 250	3 M/ESPE Dental (St Paul, MN, USA)	Tube	A2
Fuji IX	GC Corporation (Tokyo, Japan)	Powder/liquid	U
Ketac Molar	ESPE Dental AG (Seefeld, Germany)	Powder/liquid	A3
Vidrion R	SS White Artigos Dentários Ltda (RJ, Brazil)	Powder/liquid	U
Vitromolar	DFL Indústria e Comércio Ltda (RJ, Brazil)	Powder/liquid	A3

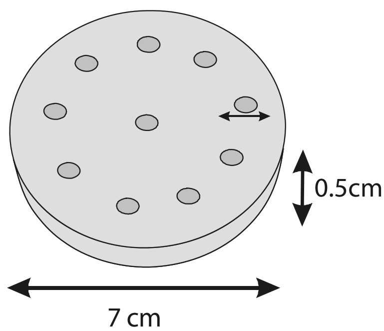

Fig. 1 Teflon mold with 10 circle perforations.

After mixing according to manufacturer instructions, cements were inserted into the mould and filled to excess. A polyester strip was used to cover the cement until 7 min of initial reaction were completed. Slight pressure was applied and the bulk of extruded excess cement was removed. Cements were covered on one side with a layer of nail varnish, and the opposite side was prepared for analysis by covering with a layer of solid vaseline, then covering with a glass plate and isolating the mould margin with wax. The wax was removed with a gauze at the moment of the test. The specimens were then transferred to an oven set at 37°C, with distilled water, and were removed from this environmental chamber only at the moment of the analyses, avoiding water loss and gain.

Vicker's microhardness measurements were taken at 1 day and 1 week after the initial setting reaction using a digital microhardness equipment/meter (Otto Wolper—Werke GMBH, Illinois, USA). Microhardness indentations were made on the top of specimens' surfaces and were divided into four quadrants. Vicker's diamond indentations were performed under a load of 100 g for 30 s [11]. Three measurements were taken in each quadrant, totaling twelve measurements. Vicker's values were converted into microhardness values by the machine.

The results were subjected to Student's T test to compare the mean values among the groups and a Tukey test was performed to investigate the interaction between materials and measurements. All statistical analyses were conducted at a confidence interval of 95%.

3 Results

The mean of Vicker's microhardness measurements taken after 1 day and 1 week storage are shown in Table 2 and Fig. 2.

The results of the statistical analysis demonstrate that composite resin microhardness presented superior values in comparison to glass ionomer cements. After 1 day, no statistical significant difference in microhardness was observed among the glass ionomer cements ($p > 0.05$). However, after 1 week, Ketac Molar, Vidrion R and Vitromar demonstrated an increase in microhardness values ($p < 0.0001$). Fuji IX presented a lower microhardness mean value than the other

Fig. 2 Graphical representation of microhardness mean values.

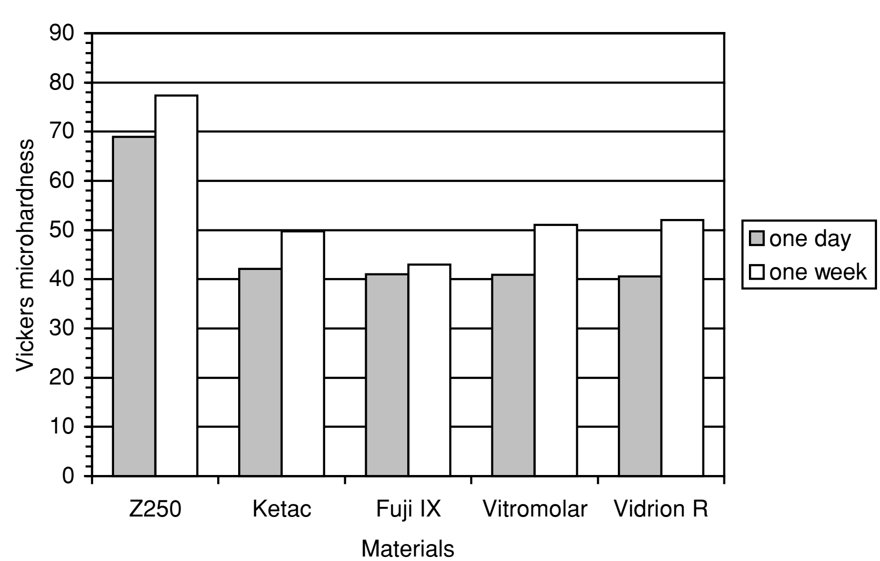

Table 2 Microhardness mean, standard deviation (SD) and p value of T Student statistical analysis of the materials in a 95% confidence interval

Material	Measurements	Mean	SD	p value
Z250	1 day	69.0	3.73	<0.0001
	1 week	77.3	5.59	
Ketac	1 day	42.1	2.30	<0.0001
	1 week	49.8	3.12	
Fuji IX	1 day	41.0	4.03	0.1400
	1 week	43.4	2.71	
Vitromolar	1 day	40.9	4.33	<0.0001
	1 week	51.1	4.11	
Vidrion R	1 day	40.6	0.77	<0.0001
	1 week	52.0	1.51	

cements, with no statistical difference when comparing their values at 1 day and 1 week.

4 Discussion

Microhardness is one of the most important physical properties of a dental material and may be defined as the resistance of a material to indentation or penetration. It depends on the interaction of several properties, such as ductility and malleability [12]. For Vicker's microhardness measurements, the hardness number increases as surface hardness increases. Change in hardness may reflect the cure state of a material and the continuation of a setting reaction [9, 10].

The results of the present study revealed no significant difference in microhardness among cements evaluated 1 day after setting, however a difference existed after 1 week (Table 2 and Fig. 2). According to some authors, for glass ionomer materials, an improvement in mechanical properties as a function of time can be verified, reflecting the continuity of the setting reaction. However, superficial microhardness cannot reliably detect the setting reaction that occurs in the bulk of the material [9, 13].

Our results demonstrated an exception to this theory; namely Fuji IX. This material presented no significant increase in microhardness, while all the other glass ionomer cements demonstrated increased microhardness at 1 week. This finding may indicate that the hardening phase of the setting reaction of the material was still occurring during day 1 after setting. This setting reaction phase occurs after the gelation phase and involves the continued formation of aluminum salt bridges [14].

The initial resistance of the glass ionomer cement has been conditioned by numerous factors such as the chemical composition, glass structure, concentration and molecular weight of the polycarboxylic acid and the proportion of powder/liquid [15]. Therefore, one possible elucidation may be related to the smaller mean particle size of Fuji IX, resulting in a greater surface area for polymeric acid and glass interaction, leading to a faster maturation rate. Another possible contributing factor may be the use of a higher powder to liquid ratio [9]. Interestingly, Vidrion R, which presented the smallest variability of microhardness values among the specimens, possibly indicating a greater homogeneity among the specimen surfaces.

The mean microhardness values for Z250 found in this study were significantly superior to those of the glass ionomers materials. Superior microhardness values for composite resins were in agreement to other studies [10, 15]. The setting reaction consists of a conversion of the double-linkage of the organic fillers, Bis-GMA (bisphenol A-diglycidyl metacrylate) and TEGDMA (triethylene glycol dimetacrylate), which provides a strong matrix [16–18].

When inserted in the oral environment, dental materials are exposed to other phenomena, not simulated in this study, which may affect microhardness. Wear and abrasion are also related to changes in microhardness levels and this property can be considered an important parameter for prediction of the clinical performance of the material [17, 19]. However, *in vitro* studies do not reflect what exactly occurs in an oral cavity and offer inaccurate information in regards to the interaction between restorative materials and oral phenomena. When inserted in the oral environment, restorative materials are exposed to saliva, pH changes and other factors such as food and liquid. Their surfaces suffer abrasion, erosion and wear caused by tooth brushing, drinks, food and mastication [20].

Consequently, the dental materials indicated for the ART approach, such as glass ionomer cements, need to have their physical properties improved to ensure their optimal long-term clinical performance.

5 Conclusion

Based on the results of the present study, we show that the glass ionomer cements evaluated demonstrated an increased microhardness 1 week after setting, with the exception of Fuji IX.

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