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A discriminatory mechanical testing performance indicator protocol for hand-mixed glass-ionomer restoratives

Mirza Shahzad Baig[a], Adam H. Dowling[a], Xu Cao[b], Garry J.P. Fleming[a,*]

[a] Materials Science Unit, Dublin Dental University Hospital, Trinity College Dublin, Dublin 2, Ireland

[b] Barts and The London School of Medicine and Dentistry, Centre of Adult Oral Health, Turner Street, Whitechapel, London E1 2AD, UK

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ABSTRACT

Objectives. To identify a reproducible and discriminatory mechanical testing methodology to act as a performance indicator for hand-mixed glass-ionomer (GI) restoratives.

Methods. Groups of 20 (five batches of four) cylinders (6.0 ± 0.1 mm height, 4.0 ± 0.1 mm diameter) for compressive fracture strength (CFS) and compressive modulus (CM) testing, bars (25.0 ± 0.1 mm length, 2.0 ± 0.1 mm width, 2.0 ± 0.1 mm thickness) for three-point flexure strength (TFS) and tensile flexural modulus (TFM) testing, discs (13.0 ± 0.1 mm diameter, 1.0 ± 0.1 mm thickness and 10.0 ± 0.1 mm diameter, 3.10 ± 0.03 mm thickness) for biaxial flexure strength (BFS) and Hertzian indentation (HI) testing, respectively, were prepared using a hand-mixed GI restorative manipulated with 100–20% (in 10% increments) of the manufacturers recommended powder content for a constant weight of liquid. Data were statistically analyzed at $p < 0.05$, the coefficient of variation (CoV) was assessed for the four tests at each powder:liquid mixing ratio investigated ($n = 9$) and a Weibull analysis performed on the CFS, TFS and BFS data to assess the reliability of the data sets. The failure mode and fracture origin of the HI specimens was assessed by fractography.

Results. For the hand-mixed GI restorative, a progressive reduction in the powder content (by 10% for a constant weight of liquid) resulted in a progressive linear deterioration ($p < 0.001$) in the CFS ($R^2 = 0.957$), CM ($R^2 = 0.961$) and TFM ($R^2 = 0.982$) data. However, no linear deterioration ($p > 0.05$) was identified for the TFS ($R^2 = 0.572$), BFS ($R^2 = 0.81$) and HI ($R^2 = 0.234$). The CoV and Weibull data identified distinct regions – three for the CFS and TFS data and two for the BFS data sets, within the range of powder:liquid mixing ratios investigated. Fractographic analysis of HI specimens revealed a transition in failure mode from bottom-initiated radial cracking to top-initiated cone cracking on reducing the powder content for a constant weight of liquid.

Significance. The CFS test is the only discriminatory performance indicator for hand-mixed GIs from amongst the four mechanical testing approaches (CFS, TFS, BFS and HI) investigated. The CM and TFM represent an intrinsic material property independent of specimen

* *Corresponding author*. Tel.: +353 1 612 7371; fax: +353 1 612 7297.
E-mail address: garry.fleming@dental.tcd.ie (G.J.P. Fleming).

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dimensions and may be used as an adjunct to a mechanical testing approach when investigating hand-mixed GIs.
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1. Introduction

The discovery of glass-ionomers (GIs) by Wilson and Kent [1] in 1969 was accomplished on testing the 200th glass composition (G-200) and a clinically acceptable dental cement was realized [2]. G-200 remained the only glass composition capable of forming a GI suitable for clinical use prior to the discovery of the role of tartaric acid in the setting reaction [3]. Following the establishment of the role of tartaric acid as a chelating agent [3], further opportunities were identified for a number of glass compositions other than G-200 [2]. Since the original ground breaking research into the development of GIs, manufacturers and investigators have employed different glass formulations to improve the mechanical properties, enhance radiopacity and increase the reactivity of the glass powder by modifying the calcium fluoroaluminosilicate glass composition [4] with strontium [5], zirconia [6], lanthanum [7] and zinc [8]. Additionally, investigators have prepared aluminoborate [9] and zincsilicate [10,11] glass compositions for use in GIs with varied levels of success.

The compressive fracture strength (CFS) testing methodology is the only strength test specified in the International Organization for Standardization (ISO-9917-2003) [12] for GIs. However, the reliability [13] and validity [14] of the CFS testing protocol have been questioned [15]. A 'test-house variability' study in 1990 [13] identified inter-operator variability (between different test centers) was considerable and intra-operator variability (between individual operators) was also significant such that the poor reliability of the CFS testing protocol was 'inappropriate' for inclusion in the standard [12]. Additionally, the failure mode of dry gypsum cylinders was 'vertical split slabbing mode' but when wet with water or alcohol changed to failure on the 'diagonal planes running from top left to bottom right' [14]. However, the stress at failure calculation was independent of the failure mechanism [14] which raised concerns regarding the validity of the CFS test [14]. Alternative mechanical testing approaches including three-point flexure strength (TFS) [16], biaxial flexure strength (BFS) [16] and Hertzian indentation (HI) [17] tests have been suggested as potential replacement testing methodologies to be adopted in ISO-9917-2003 [12] – albeit for encapsulated GIs. Encapsulated GIs offer control over inaccurate dispensation of the powder and liquid components by the operator, where the optimum powder and liquid proportions are predetermined by the manufacturer [18,19]. The issue of identifying a reliable [13] and valid [14] testing methodology is complicated further for hand-mixed GIs as a result of the range of powder:liquid mixing ratios routinely employed clinically [20]. Interestingly, powder contents as low as 37% of that specified by the manufacturer were identified for Chemfil II, a hand-mixed GI, when prepared by 22 dental nurses – eleven employed in general dental practice and eleven from a dental hospital setting [20]. Hand-mixed GIs tested in the laboratory are prone to large variations in the mechanical property data arising from inadequate control of the testing conditions [16] and the non-standardized nature of the product delivery systems namely powder scoops [20,21] and liquid dropper bottles [22,23]. The microstructure of a set GI is composed of unreacted glass core fillers sheathed by a siliceous hydrogel and dispersed in a polyacrylate matrix [24–27]. Therefore, the mechanical properties of GIs arise from the reinforcing glass phase where a linear deterioration on reducing the powder content from that specified by the manufacturer would be expected following testing with an appropriate testing methodology [28,29].

It is important to appreciate, however, that if one was charged with the task of delivering a novel reinforced GI restorative, the optimum powder:liquid mixing ratio [30] would need to be identified by hand-mixing the GI liquid with varying GI powder content and performing preliminary mechanical tests. The mechanical testing protocol of choice would therefore require to be discriminatory between the different powder:liquid mixing ratios employed, thereby identifying a linear deterioration when the powder content is reduced from the optimum value. As a result, the aim of the current study was to identify a reproducible and discriminatory testing methodology to act as a performance indicator for hand-mixed GI restorative materials. The hypotheses proposed was that each of the commonly used mechanical testing protocols employed to determine the performance of GIs (CFS, TFS, BFS and HI) would act as performance indicators by identifying the linear deterioration in the mechanical properties on reducing the powder content from that specified by the manufacturer.

2. Materials and methods

The GI restorative used (Ionofil Molar: Voco GmbH, Cuxhaven, Germany, Powder: V50335, Liquid: V50375) was hand-mixed for 40 s with the manufacturer's recommended powder:liquid mixing ratio (4:1 g:g) [31]. A glass slab was placed on a balance accurate to 0.001 g (Sartorius Expert, Sartorius AG, Goettingen, Germany), the appropriate weight of GI powder and liquid were dispensed and the powder divided into two equal portions. A stainless steel mixing spatula was used to mix half the powder with the liquid for 20 s, followed by the addition of the remaining powder for a further 20 s in a temperature (21 ± 1 °C) and humidity (50 ± 5%) controlled laboratory [31]. To simulate the distribution of powder:liquid mixing ratios routinely produced by operators in clinical practice, GIs were prepared by reducing the manufacturers recommended powder content (in 10% increments for a constant weight of liquid) – to a minimum of 20%.

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