Original Article

Physico-mechanical properties of a nanofilled glass ionomer cement

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ABSTRACT

Background: The use of Glass ionomer cements (GIC) as restorative materials is beneficial due to fluoride release and ease of application. Strength and solubility are important properties that can affect the longevity of restorative materials in the oral environment.

Aim: The objective of this study was to evaluate compressive, diametral tensile strength and solubility of a nanofilled GIC (Ketac N100) compared to available GICs, Fuji IILC and Fuji IX.

Material &Methods: Compressive and diametral tensile strength were tested by constructing 10 samples (6mm length x 4mm diameter) then tested as specified by the International Organization for Standardization (ISO 9917-2003). Solubility was assessed by constructing five samples (20mm diameter x 1.5mm thick), then storing them in distilled water for 24 hours before testing. Solubility was measured by weighing the residue that remains of each material following water evaporation of a portion of the suspension solution and calculating solubility as the total amount of soluble component in ratio to the total initial weight. Data were analyzed using one-way analysis of variance (ANOVA) and Tukeys test (*p≤0.05*).

Results: Compressive and diametral tensile strength of Ketac N100 was significantly lower than that of Fuji II LC (p<0.05). The 24 hour solubility of Ketac N100 was significantly higher than Fuji IX and Fuji II LC (p<0.05).

Conclusions: This study suggests that the 24 hour strength of nanofilled GIC was inferior to that of conventional resin-modified GIC, in addition to showing higher solubility.

Keywords: Resin modified Glass ionomer cement, Mechanical properties, Nanofilled glass ionomer cement, Solubility, Strength.

INTRODUCTION

The wide spread use of Glass ionomer cements (GIC) in dentistry since they were introduced by Wilson and kent [1] has been well documented due to their several advantages and desirable properties. These include biocompatibility, adhesion to moist enamel and dentin and anticariogenic ability due to fluoride release [1-3]. The increased popularity of GICs has led to their use as restorations, liners and bases, luting agents and pit and fissure sealants [4, 5].

The extensive use of GICs has been accompanied by numerous laboratory studies conducted to investigate physical, chemical and biological properties of the various formulations available in the market [6-9]. However, their brittleness, poor esthetics, low compressive and tensile strength, and sensitivity to moisture in the early stages of the setting reaction, placed limitations on their use as restorative materials [10, 11].

Conventional GICs have been modified by the incorporation of a resin monomer which yielded the so called resin modified GICs that set partly via an acid/base reaction and partly through photochemical polymerization [10]. The materials were introduced, partly, to overcome moisture and dehydration sensitivity of the early versions of the glass ionomers, in the initial setting stages, and low early mechanical strengths associated with conventional GICs, while maintaining their clinical advantages [12, 13]. The advantages and various clinical application of resin modified GICs have been well-established over the past years as can be seen from their widespread use by dentists [6].

A new technology aiming at improving physical properties of GICs has been introduced in the form of incorporating nano-filler particles into resin modified GICs (nano-RMGI; Ketac N100, 3M-ESPE, Seefeld, Germany). Its primary curing mechanism is by light activation. The main advantages of such modification include, increased

ability of filler packing, improved mechanical properties and improved polish and esthetic properties as claimed by the manufacturer [6, 14].

As mentioned earlier, inferior mechanical strength in addition to relatively high solubility of conventional GICs has lead to the introduction of improved and modified formulations of this material including nano-filler incorporation. Therefore, laboratory based studies are required to investigate this novel technology and its' effect on the modified material. A wide range of mechanical properties are commonly used to characterize different formulations of GICs such as compressive strength, diametral tensile strength, fracture toughness and microhadness [10, 11, 14, 15].

No data is currently available on the compressive and diametral tensile strength of Ketac N100 or its solubility to the best knowledge of the authors of the current study. Despite the fact that Ketac N100 is marketed by the manufacturer as an esthetic GIC to be used in low stress bearing area, testing mechanical properties of GICs used for different clinical applications is commonly done [15], in addition to the fact that Ketac N100 has been recommended for use as a base underneath restorations as it might improve fracture strength of endodonticaly treated teeth and to improve marginal seal [16, 17]. Consequently, the aim of the current study is to assess the compressive, diametral tensile strength and 24 hour solubility of a nanofilled GIC in comparison to conventional and resin modified GICs.

METHODS

The materials and manufacturer of the tested materials in this study are shown in table 1. Specimens of GIC were made, and physical/mechanical properties were tested following the International Organization for Standardization (ISO) Standard 9917:2003 dental water-based cements [18] for the compressive and diametral tensile strength tests, as outlined in the following sections. The solubility measurements were done according to the methodology described by Peez & Frank [8]. Resin modified GIC samples were irradiated for 40 second on all surfaces to ensure proper setting using an LED light curing unit (Rolence Ultra-lite 500E. Rolence Enterprise Inc, Taiwan). The light was tested for output intensity (600 mW/ cm^2) using a dental radiometer (Demetron L.E.D. Radiometer, Kerr, Orange, Calif).

Ten cylindrical specimens 4mm diameter (D) × 6mm long (t) were made using split plexiglass molds. One hour after the end of mixing of Fuji IX and 15 minutes after the end of mixing of Fuji II LC

and Ketac N100, the ends of each sample were ground flat using wet 600 grit silicon carbide paper, then removed from the molds and stored in 100% RH for 24 hours prior to testing. The specimens were tested using a Computer Control Electromechanical Universal Testing Machine (model No. WDW-20, Jinan Testing Equipment, IE Corporation, China) at a crosshead speed of 1 mm/min. Each specimen was placed with the flat ends between the platens of the testing machine, and compressive load was applied along the long axis of the specimen. The compressive strength (C), measured in Mega Pascal (MPa), was calculated using the following formula: $C=4P/\pi D^2$ (where Pis the maximum force applied in Newton (N)).

Table 1: Materials used in the study

Material	Manufacturer and Description
GC Fuji IX GP	G.C. Corporation, Tokyo, Japan
	Conventional , capsulated glass ionomer cement
GC Fuji II LC	G.C. Corporation, Tokyo, Japan
	Resin modified , capsulated glass ionomer cement
Ketac N100	3M ESPE, Seefeld, Germany
	Naofilled, hand mixed glass ionomer cement

Ten cylindrical specimens were made using the same dimensions as for the compressive strength test and stored under the same conditions. After 24 hours, a compressive load was applied across the diameter of each specimen, producing a tensile stress perpendicular to the axis of the loading. Load was applied at a crosshead speed of 1 mm/min, and recorded until the specimen fractured. The tensile stress (T) measured in MPa was calculated by the following formula: $T=2P/\pi Dt$.

For solubility measurements five specimens with a diameter of 20mm and a thickness of 1.5mm were prepared for each material with a ring-shaped mould under conditions as described above. For suspension of molds in distilled water, a small hole was drilled peripheral to the sample hole. One hour after the end of mixing of Fuji IX and 15 minutes after the end of mixing of Fuji II LC and Ketac N100, the samples were thoroughly cleared of material excess and ground using wet 600 grit silicon carbide paper to remove excess material and unpolymerized surface layer or defects. Samples were then weighed using a digital analytical scale. Specimens of each tested material were immediately stored in 50 ml distilled water at 36°C. After 24 hours of storage in solution, a portion of 40ml of the eluate was taken and the water of this portion was evaporated at 120°C [8]. The residue

was weighed and solubility calculated as the total amount of soluble component in ratio to the total initial weight of the specimens expressed in percent. This value was reported as 24 h solubility.

The results of compressive and diametral tensile strength and solubility were analyzed using one-way analysis of variance (ANOVA) and Tukeys test (p≤0.05) by means of statistical software (SPSS for Windows, Release 6.1.2, IBM SPSS, Armonk, N.Y.)

RESULTS

Table 2 shows the mean values of physical properties tested following 24 hours immersion in distilled water. The compressive strength of Ketac N100 was significantly lower than that of Fuji IX and Fuji II LC (p = 0.009, 0.000 respectively).

Table 2: Mean values and standard deviation of compressive strength, diametral tensile strength and solubility for the tested materials

Property	Fuji IX	Fuji IILC	Ketac N100
	Mean (SD*)	Mean (SD)	Mean (SD)
Compressive strength (MPa)	126.2 (16.78)	146.7 (19.20)	94.2 (28.6)
Diametral tensile strength (MPa)	5.7 (1.7)	14.3 (2.9)	10.1 (3.7)
Solubility (%)	0.11 (0.02)	0.12 (0.1)	1.5 (0.1)

*Standard Deviation

There was no significant difference in the compressive strength values between Fuji IX and Fuji II LC. The diametral tensile strength of Ketac N100 was significantly higher than that of Fuji IX but significantly lower than Fuji II LC (p= 0.007, 0.009 respectively). The diametral tensile strength of Fuji II LC was significantly higher than Fuji IX (p= 0.000). Assessment of the 24 hour solubility in water of the three tested materials showed that the solubility of Ketac N100 was significantly higher than Fuji IX and Fuji II LC (p=0.000). There was no significant difference in the solubility values of Fuji IX and Fuji II LC (p= 0.957).

DISCUSSION

The glass ionomer cement (GIC) system described by Wilson and Kent [1] (1972) was introduced as a new translucent dental material that hardens as a result of the reaction between acrylic acid and alumino-silicate glass powder. GICs are formed by an acid-base reaction that occurs when polyacrylic acid comes into contact with an alumino-silicate glass. The properties of GICs depend on their chemical composition, size and amount of the glass powder filler particles, amount, concentration and molecular weight of the polyacrylic acid, in addition to the powder: liquid ratio [19].

The results of the current study show that there was a significant difference in the strength and solubility of the nanofilled GIC (Ketac N100) compared to the conventional and resin modified GICs tested. Both compressive and diametral tensile strength values of Ketac N100 were significantly lower than that of Fuji IILC. Several studies investigated physical and mechanical properties of both conventional and resin modified glass ionomer [8, 20, 21]. However only a few investigated mechanical properties of nanofilled GICs, with the majority of studies mainly investigating properties such as fluoride release and recharge, bond strength to tooth structure and surface roughness and hardness [6, 7, 9, 14].

The published data on the physical and mechanical properties of Fuji IX and Fuji IILC are variable due to the different mixing methods of these cements, variable sample dimensions, and testing method. The compressive strength of Fuji IX and Fuji IILC in the current study was 126.18 MPa and146.76 MPa respectively and the diametral tensile strength was 5.7 MPa and 14.3 MPa respectively. Both strength values for both materials are lower than values reported in previous studies [8, 21, 22] due to the reasons mentioned above. The lower strength values of Ketac N100 compared to Fuji IILC may be due to the higher filler content in Fuji IILC which was reported to be 76.2% mass fraction compared to 69% for Ketac Nano. Another possible reason is the difference in the resin matrix composition, namely, the presence of 2-hydroxyethyl methacrylate in Fuji IILC as reported previously which is not the case for Ketac Nano [21]. Furthermore, a study by Xu and Burgess [21] (2003) investigating compressive strength and fluoride release of conventional and resin modified GICs, compomers and composites suggested that the difference in physical and mechanical properties of conventional and resin modified GICs may be due to the composition of the filler [21] which may affect the strength and solubility of the materials.

The solubility of GICs has been previously investigated [8, 20]. The 24 hour solubility of Fuji IX reported by Peez and Frank [8] (2006) was 0.05 %. In another study, Fuji IILC was reported to have a solubility of 2.9 µg/mm^3 which was higher compared to the other tested materials [20]. Nonetheless, comparison of results is difficult due to different time periods and using different measurement units [20]. Moreover, Fuji IILC used in the current study was capsulated rather than hand mixed unlike the previously mentioned study. The incorporation of air

voids during manual mixing leads to the incorporation of bubbles resulting in voids which contribute to more solubility and lower strength [23]. In the current study, Ketac N100 is hand mixed then loaded in the delivery tip provided by the manufacturer to express the cement using the piston. This may have lead to the incorporation of bubbles leading to higher solubility and lower compressive strength. It was also noted by the authors that Ketac N100 was more viscous compared to Fuji IX and Fuji IILC which may also lead to more bubble incorporation during mixing. Other factors that influence solubility include filler concentration and mean particle size, particle surface area and particle type [24].

Care should be taken when results are extrapolated to the oral environment where several factors that can affect physical properties of dental materials cannot be reproduced in vitro such as saliva, chemistry of the oral environment and limitations to proper curing of restorative material [25]. Future research is needed to test materials over longer time periods, taking into consideration storage temperature, storage media in addition to comparison with other brands and resin systems.

Within the limitation of this study, it can be concluded that:

- Nanofilled GIC had inferior compressive and diametral tensile strength compared to conventional resin modified GIC.
- Nanofilled GIC showed higher solubility compared to conventional GIC and conventional resin modified-GIC used in the current study.

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