Malaysian Dental Journal (2012) Volume 34 Issue 1
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MALAYSIAN DENTAL JOURNAL

Effect of Porosity on Compressive Strength of Glass Ionomer Cements

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ABSTRACT

Purpose. The purpose of this in vitro study was to determine the correlation between the porosity of diameter (1-100) µm and the compressive strength of glass ionomer cements.

Materials And Methods. A total of fifteen cylindrical specimens 6mm height and 4mm in diameter were prepared for each type of luting cements which are Fuji I (GC Corp, Tokyo, Japan), Fuji I CAPSULE (GC Corp, Tokyo, Japan), were stored in distilled water at 37°C for 24 hours. The compressive strength for each cement type was determined The fractured surfaces of 10 randomly selected specimens for each cement type were examined using Scanning Electron Microscopy at 250 times magnification, and five photomicrographs were taken at five random places for each fractured surface. All the photomicrographs were analyzed using image analyzer software to determine the amount and size of porosity present. The data were analyzed Mann-Whitney tests for compressive strength and percentage of porosity, Spearman's rho test was used to examine the correlation between the compressive strength and the percentage of porosity (1-100) µm in diameter.

Result. Mechanical mixing method had no effect on the compressive strength, but it had a significant effect by increasing the percentage of porosity of diameter (1-100) µm in diameter of GIC. There was no linear relationship between compressive strength and porosity (1-100) µm in diameter for both type of luting cements ($P>0.05$).

Conclusion. Mixing method has no effect on the compressive strength. The size and number of the porosity in the specimens of encapsulated cements were greater than those of hand-mixed cements. The porosity (1-100) µm in diameter and the compressive strength bore no linear relationship to each other.

Key Words: Mechanical tests, Glass ionomer cement, Luting materials, Porosity.

Please cite this article as: Ali Al-Kadhim AH, Abdullah H, Mahmood A. Effect of Porosity on Compressive Strength of Glass Ionomer Cements. Malaysian Dental Journal 2012; 34(1): 23-29.

INTRODUCTION

The term "cement" implies the material will be used to lute or glue things together. While other uses are common, use as a luting agent has given this group of materials their name, cements. Dental cements retain appliances and restorations in place with macromechanical and micromechanical retentions. Some dental cements are adhesive via chemical bonds, but most are not[1,2] Luting agents comprise a broad category of materials used to attach and seal dental restorations and prosthesis to teeth. New luting agents, particularly with adhesive capability, are being introduced in an attempt to improve clinical success. The choice of luting agents is dependent on the clinical situation combined with the physical, biological, and handling properties of the luting

Agent.[3,4] Caries and crown dislodgment are the most common reasons for failure of crown and bridges. Caries may also relate to cement micro fracture and consequent micro leakage; the dislodgment may be related directly to gross mechanical failure of luting cements.[3,5] The properties of luting materials in general can be divided into mechanical properties which are evoke by the application of mechanical forces and physical properties which do not involve application of mechanical forces.[6] In recent years, the most common water-based cements used for final cementation of crowns and bridges are glass ionomer cements.[7] Various techniques have been

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used to understand the complex microstructure of glass ionomer cement, including chemical analysis, optical microscopy, infrared spectroscopy, electron microscopy (both transmission and SEM), and x-ray microanalysis.

Each of these techniques has contributed to understanding of the setting reaction, composition, and microstructure of glass ionomer cements.[8] Several workers have reported that mechanical properties of encapsulated materials were inferior or equivalent to those of the hand-mixed materials. It was recognized that mechanical mixing may result in the incorporation of air porosity in the cements, leading to weakening.[9]

The presence of pores or voids in luting cements may affect the cements in a number of adverse ways; particularly where the lute is exposed to the oral cavity at the margins of cemented restorations, the presence of pores will increase the likelihood of mechanical failure.[10,11] If the porosity in dental luting cements is to be minimized, a better understanding of the nature and origin of such voids is necessary. In particular, information is required about the number, size and morphology of such pores, which will determine the extent of their likely adverse effects. Furthermore, knowledge of the origin of such porosity might be facilitated by characterizing the pattern of voids 9. The objectives of this study are to:

Measure the percentage of porosity of diameter (1-100) µm at the fracture surface in resin adhesive cement, glass ionomer cement (hand-mixed and encapsulated). Determine the correlation between porosity of diameter (1-100) µm at the fracture surface and the compressive strength of luting cements.

MATERIALS AND METHODS

Two types of luting cements were used in this study ,which are hand-mixed conventional glass ionomer luting cement namely, Fuji I (GC Corporation, Tokyo, Japan), and encapsulated conventional glass ionomer luting cement namely, Fuji I CAPSULE (GC Corporation, Tokyo, Japan). Each type of luting cement consisted of fifteen specimens stored in distilled water for 24 hours at 37 °C with at least 90% relative humidity. All the materials were mixed according to the manufacturer’s instructions. The handmixed conventional glass ionomer cement Fuji I (one scoop of powder and two drops of liquid) was mixed with a plastic spatula and a paper pad for 20 seconds. The encapsulated glass ionomer cement (Fuji I CAPSULE) was mixed by rotating using RotoMix, (3M, EPSE, Seefeld, Germany) for 10 seconds without centrifuge as recommended.

Specimen preparation

The following procedures were performed in a room at 23 ºC. The humidity was not controlled but was around 50% RH. Specimens were prepared and the testing was conducted by single person to maximize the standardization. Specimens of each material were prepared in a similar manner. Thirty test specimens were prepared in a cylindrical poly tetra fluroethylene split moulds, with internal dimensions 6 mm ± 0.1 mm high and 4 mm ± 0.1 mm diameter as shown in figure 1.

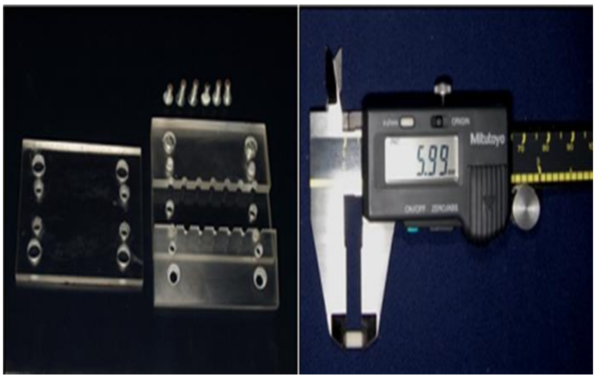

Figure 1. Teflon split mould and micrometer screw gauge (Kawasaki, Japan)

Within 60 seconds after the end of mixing, a slight excess of the mixed luting cement was placed into the mould, which was resting on a polyester strip in order to prevent the adhesion of poly-acrylic acid-based cements. For the encapsulated luting cements, the nozzle of the capsule was inserted into the cavity of the mould and touched to the wall of the mould. The nozzle was raised up slowly as the mould was filled as as shown in figure 2.

One hundred and eighty seconds after the end of the mixing, the whole assembly of the specimens and mould was placed in an environmental chamber (incubator) at 37 ºC and relative humidity of at least 90%, for one hour. Exactly one hour after placing in the incubator the plates were removed and the end of the specimens were grinded flat at right angle to its long axis by using 800-grit silicon carbide paper under continuous water irrigation by using a Twin Wheel Grinder/Polisher machine (Buehler Uk, Conventry. England). The specimens were checked visually without magnification for air voids or chipped edges, all the defected specimens were discarded. And in order to facilitate the removal of the hardened cement specimens, the internal surfaces of the mould were evenly coated by paraffin wax. The luting cements specimens were carefully removed from the moulds and then stored in distilled water in environmental chamber at 37 ºC for 23 hours.

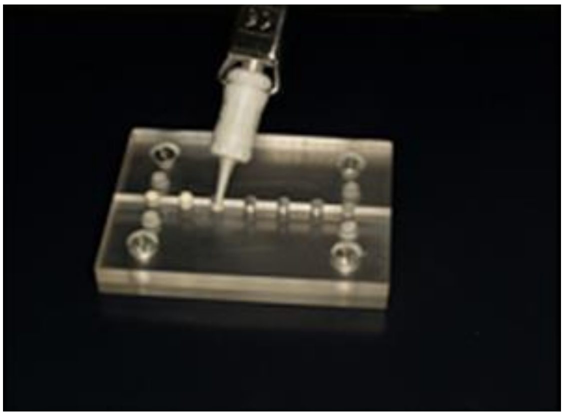

Figure 2. The nozzle of the capsule was inserted into the cavity of the mould and touched to the wall of the mould

Evaluation of compressive strength

The Universal testing machine which used in this study was SHIMADZU (SHIMADZU Corporation, Kyoto, Japan).The diameter of the specimens was measured with a micrometer screw gauge (Kawasaki, Japan) accurate to 10 µm. The flat ends of the specimens were covered with a wet piece of filter paper to ensure the specimens were tested "wet" and a compressive load applied, with a cross head speed of 0.5 mm/min to the long axis of the specimens. The maximum load to failure was recorded and the procedure repeated so that the minimum of 15 nominally identical standard cylindrical specimens had been fractured for each type of luting cement.

Porosity evaluation

After the compressive strength evaluation, fracture surface of one fragment which was randomly selected from ten randomly selected specimens for each group were examined by SEM (XL 40 series, PHILIPS, Holland). The specimens were sputter coated with gold prior to SEM examination and the fracture surfaces were observed at an operating voltage of 3kV. Photomicrographs were taken by scanning electron microscope at X250 original magnification. SEM imaging was done on low vacuum mode. Five micrographs were taken for each fracture surface at random places in order to determine the percentage of porosity of diameter (1-100) µm at the fracture surfaces by using the Direct Counting Method.

Direct counting method:

Direct counting method was used in this study to measure the percentage of porosity at the fracture surface; this method has the advantages of being reliable and simple and showed no difference as compared with the Point counting method.[12,13] The disadvantage is that all the pores are assumed to be spherical in shape.

The direct counting method categorized the pores into four categories according to its diameter which is <1µm, (1-10) µm, (10-50) µm, (50-100) µm, and because pores of diameter less than 1µm have no effect on the mechanical properties of luting cements,[14] this category of pores was eliminated from this study.

All pores in different size ranges were identified and the longest pore diameter measured within photomicrographs with a digital micrometer (Image Analyzer Software, Leica QwinLite, Leica Microsystems imaging solution Ltd., Cambridge, UK). The percentage of porosity for each size range within a given measurement area (NA) was calculated by the following formula:

Ps = (nr2π /NA) X 100

Where

Ps - percentage of porosity for each size interval.

n - number of pores

r - radius of pores.

Na - measurement area.

The diameter measurement of the pores for each photomicrograph was repeated twice by the same operator within 3 days interval between each readings, by using the image analyzer, the means value were taken to measure the percentage of porosity by using the direct counting method.

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RESULTS

Compressive strength
Because all the values were not normally distributed according to the bell curve, Mann-Whitney U test was used to compare the compressive strength between groups, the level of significance was set as $P<0.05$. It showed that there was no statistically significant difference ($P>0.05$) between the compressive strength of Fuji I and Fuji I CAPSULE.

Compressive strength Median (IQR)		P value
Fuji I	Fuji I CAPSULE	
154.587 (58.164)	162.975 (78.045)	1

Porosity
The objective was to compare the percentages of porosity (1-100) µm in diameter between both types of luting cements. Porosity was categorized with respect to the diameter in three categories, porosity (1-10) µm, (10-50) µm, (50-100) µm. MannWhitney test was used to compare the percentage of porosity of diameter (1-10) µm between groups, the level of significance was set as $P<0.05$. It showed that the percentage of porosity (1-10) µm in diameter of Fuji I CAPSULE was highly significant ($P<0.05$) as compared with Fuji I.

Porosity (1-10) µm in diameter Median (IQR)		P value
Fuji I	Fuji I CAPSULE	
0.0450 (.060)	0.1350 (.0875)	0.001

While for the percentage of porosity of diameter (10-50) µm between groups, showed that there was no statistically significant difference ($P=0.162$) between the percentage of porosity (10-50) µm in diameter of Fuji I and Fuji I CAPSULE.

Porosity (10-50) µm in diameter Median (IQR)		P value
Fuji I	Fuji I CAPSULE	
1.8700 (1.120)	2.0700 (1.220)	0.162

On the other hand for the percentage of porosity (50-100) µm showed that there was no statistically significant difference ($P=1.00$) between the percentage of porosity (50-100)µm of Fuji I and Fuji I CAPSULE.

Porosity (50-100) µm in diameter Median (IQR)		P value
Fuji I	Fuji I CAPSULE	
2.2100 (2.190)	1.5300 (2.460)	1.00

Correlation between compressive strength and porosity
The objective was to determine if there is any correlation between the compressive strength and the incorporated porosity for each porosity category i.e. if there was any linear relationship between compressive strength and percentage of porosity (1-10) µm, (10-50) µm, (50-100) µm in diameter. Spearman's Test was used to determine the relationship between the compressive strength and the percentage of surface area porosity at the fracture surface for both type of glass ionomer cements. For Fuji I (hand-mixed glass ionomer cement), there was a strong linear relationship between compressive strength and porosity (50-100) µm in diameter with P value equal to 0.026, while there was no relationship between compressive strength and porosity (1-10) µm, (10-50) µm in diameter with a P values ($P=0.116$), ($P=0.200$) respectively. For Fuji I CAPSULE, there was no relationship between the compressive strength and porosity (1-10) µm, (10-50) µm, (50-100) µm in diameter. The P values were ($P=0.637$), ($P=0.855$), ($P=0.725$) respectively.

DISCUSSION

The most common and useful mechanical properties for characterizing luting cements are compressive strength and flexural strength.[5] Compressive strength has been considered as a critical indicator for the success of the luting cements because high compressive strength is necessary to tolerate the masticatory forces.[1,11]

Correlation between compressive strength and percentage of porosity of diameter (1-10) µm using Spearman's test		
Group	r	P value
Fuji I	0.529	0.116
Fuji I CAPSULES	0.171	0.637

Correlation between compressive strength and percentage of porosity of diameter (10-50) µm using Spearman's test		
Group	r	P value
Fuji I	-0.442	0.200
Fuji I CAPSULES	-0.067	0.855

Correlation between compressive strength and percentage of porosity of diameter (50-100) µm using Spearman's test		
Group	r	P value
Fuji I	-0.693	0.026*
Fuji I CAPSULES	-0.128	0.725

In this present study a Teflon split mould which is capable of holding a maximum of seven samples were used to fabricate the specimens which are in accordance with the ISO 9917:1991(E) for water based cements. This method appears to be sensitive to distinguishing changes in mechanical properties of brittle materials through changes in composition and level of porosity.[15,16]

The methods used in this present study to determine the compressive strength were similar to the previous study, [9,15,19] which was according to ISO 9917:1991 (E) for water-based cements. This method appears to be sensitive to distinguishing changing in mechanical properties of brittle materials through changing in composition and level of porosity.

Pores act as a source of stress concentration area, thus, making the specimen more brittle.[18] In this study the percentage of porosity (1-100) µm at the fracture surfaces of ten randomly selected specimens, of each luting cements=, was carried out by using scanning electron microscopy. The photomicrographs of SEM were analyzed by using the Image Analyzer. In order to decrease the amount of bias in the results of porosity determination on the fracture surface of each specimen, one fragment of the fractured specimens after the compressive strength test was chosen randomly and five photomicrographs of SEM were taken in five random different areas.

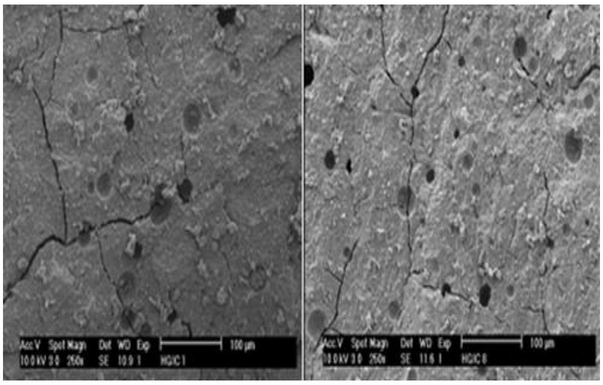

Figure 3A. Scanning electron microscopy photo-micrograph showing the porosity at the fracture surface of Fuji I

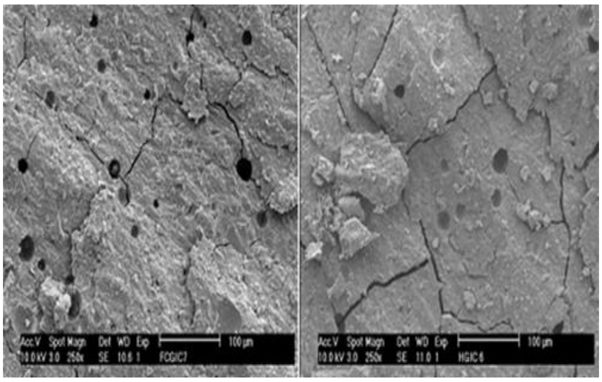

Figure 3B. Scanning electron microscropy photo-micrograph showing the porosity at the fracture surface of Fuji I CAPSULE

Nomoto and McCabe (2001) showed in their study results that, compressive strength of hand-mixed glass ionomer cement is higher and statistically significant than that of encapsulated one.

In this present study showed the compressive strength of hand-mixed glass ionomer cement (Fuji I) was higher than that of encapsulated glass ionomer cement (Fuji I CAPSULE), but when Kruskal Wallis test was used to compare the compressive strength, the P value was more than 0.05, thus there was no statistical difference between them, this could be related to the powder and liquid of the hand-mixed glass ionomer cement was not weighted during the fabrication of the specimens, just followed the manufacturer's instructions of one scoop of powder and two drops of liquid in order to be more clinical relevant, and because the amount of liquid vary depending on the inclusion of air bubbles, and the manner in which the bottle is held and the drop dispensed, and the volume of powder vary due to the differences in cement powder packing densities on filling the scoop,[15,19,20] because the powder/liquid affect the compressive strength and powder and liquid ratio which was used in clinical practice are often much lower than those recommended by the manufacturer's instructions,[21] moreover, Nomoto and McCabe used different brand of glass ionomer cements.

The standard deviation for the encapsulated glass ionomer cement was not lower than that of hand-mixed glass ionomer; this indicates that the encapsulated cement had no advantages in a term of reproducibility over hand-mixed materials.[22] This study showed that small pores of diameter (1-10) µm were present throughout the whole materials, and larger air bubbles (50-100) µm were less enormous and scattered intermittently. This result is in agreement with other studies.[8,9,16,23]

This present study showed that the encapsulated glass ionomer cement has more pores with diameter (1-10) µm than that of handmixed glass ionomer cement and was statically significant with P value less than 0.05. This could be related to the rapid mixing process of the mechanical mixing which cause air inclusion, and slower mixing of hand-mixing procedure in which the material is spatulated helps to avoid these inclusions and may also collapse some air bubbles,[9] while for the percentage of porosity of diameter (10-50) µm and (50-100) µm, there were no statically significant differences with P value more than 0.05 between them.

This result is in agreement with other studies,[9,16] they found that more bubbles were produced during mechanical mixing. The size, number and total volume of bubbles in the specimens of encapsulated cement were greater than those of the hand mixed cement. They also showed that there was a strong linear relationship between the mean of compressive strength and the mean of porosity in convention glass ionomer luting cement, and they claimed that mixing method had minimal effect on the porosity and compressive strength for the this type of luting cement, and they didn't mention the diameter of pores that bore strong linear relationship with the compressive strength.[9]

CONCLUSION

Mixing method had no effect on the compressive strength of conventional glass ionomer cements. Encapsulated glass ionomer luting cements (Fuji I CAPSULES) contained more air bubbles than the equivalent hand-mixed glass ionomer luting cement (Fuji I), Porosity of diameter (1-100) µm had no effect on the compressive strength except for hand-mixed glass ionomer luting cement (Fuji I) which showed a strong linear relationship between compressive strength and porosity of diameter (50-100) µm.

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