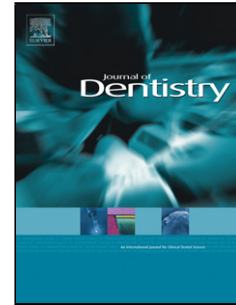


## Accepted Manuscript

Title: Improving the standard of the standard for glass ionomers: an alternative to the compressive fracture strength test for consideration?

Authors: Adam H Dowling, Garry JP Fleming, Owen Addison



PII: S0300-5712(11)00308-3  
DOI: doi:10.1016/j.jdent.2011.12.002  
Reference: JJOD 1812

To appear in: *Journal of Dentistry*

Received date: 13-8-2011  
Revised date: 7-11-2011  
Accepted date: 1-12-2011

Please cite this article as: Dowling AH, Fleming GJP, Addison O, Improving the standard of the standard for glass ionomers: an alternative to the compressive fracture strength test for consideration?, *Journal of Dentistry* (2010), doi:10.1016/j.jdent.2011.12.002

This is a PDF file of an unedited manuscript that has been accepted for publication. As a service to our customers we are providing this early version of the manuscript. The manuscript will undergo copyediting, typesetting, and review of the resulting proof before it is published in its final form. Please note that during the production process errors may be discovered which could affect the content, and all legal disclaimers that apply to the journal pertain.

Abstract: Objectives: Three strength tests (compressive, three point flexure and biaxial) were performed on three glass ionomer (GI) restoratives to assess the most appropriate methodology in terms of validity and reliability. The influence of mixing induced variability on the data sets generated were eliminated by using encapsulated GIs. Methods: Specimen groups of 40 (eight batches of n=5) cylinders (6.0±0.1 mm height, 4.0 ±0.1 mm diameter) for compressive testing, bars (25.0±0.1 mm length, 2.0±0.1 mm height, 2.0±0.1 mm width) for three point flexure testing and discs (13.0±0.1 mm diameter, 1.0±0.1 mm thickness) for biaxial testing were randomly prepared by an operator. The strength data sets for each GI restorative were pooled and one-way analyses of variance (ANOVAs) were conducted to compare between GI restoratives (p=0.05). The coefficient of variation (CoV) values for each test were pooled and a one-way ANOVA was conducted to test for differences between the reliability of the three tests.

Results: For the GI restoratives, the one-way ANOVA showed significant differences when tested in compression (p=0.001) but not when tested in three point (p=0.271) or biaxial (p=0.134) flexure. The pooled CoV values showed no significant difference between the three strength tests (p=0.632).

Conclusions: The compressive fracture strength test specified for GIs in the International Organisation for Standardisation (ISO 9917-1: 2003) should be replaced and should no longer be advocated for the predictive performance modelling of GI restoratives.

Improving the standard of the standard for glass ionomers: an alternative to the compressive fracture strength test for consideration?

Adam H Dowling<sup>1</sup>, Garry JP Fleming<sup>1</sup> and Owen Addison<sup>2</sup>.

<sup>1</sup>Materials Science Unit, Division of Oral Biosciences, Dublin Dental University Hospital, Trinity College Dublin, Lincoln Place, Dublin 2, Ireland and <sup>2</sup>Biomaterials Unit, University of Birmingham School of Dentistry, St Chads Queensway, Birmingham B4 6NN, UK

Short Title: Glass ionomer restoratives.

Keywords: glass ionomer restorative, ISO 9917-1: 2003, compressive fracture strength, three point flexure strength, biaxial flexure strength, validity, reliability.

Corresponding Author: Garry JP Fleming,  
Materials Science Unit,  
Division of Oral Biosciences,  
Dublin Dental School & Hospital,  
Trinity College Dublin,  
Lincoln Place,  
Dublin 2.

Telephone: 00 353 1 612 7371

Fax: 00 353 1 612 7297

E-Mail: [garry.fleming@dental.tcd.ie](mailto:garry.fleming@dental.tcd.ie)

## Introduction

The only strength test specified for glass ionomers (GIs) in the International Organisation for Standardisation (ISO 9917-1: 2003 [1]) is the compressive fracture strength (CFS) test although the validity [2-3] and reliability [4] of the CFS testing methodology have been challenged in the literature. In Part 1, “The crushing truth about glass ionomer restoratives: exposing the standard of the standard” [5], the authors determined experimentally the reliability of CFS data of three GI restoratives generated in accordance with the ISO testing protocol (ISO 9917-1: 2003 [1]). In CFS determination, the use of batch-censoring [1] was considered unsafe as it misidentified operative variability [5]. The stress at failure calculation neglects the failure mechanism [2] whereby uniaxially compressed cylinders collapse due to ‘some unresolved combination of tension and shear’ [2] stresses. Therefore, CFS data interpretation was limited to a measure of the ‘quality of the cement’ rather than a ‘predictive value’ [3].

It is over 20 years since the original publication by McCabe et al. [4] questioned the validity of the CFS test and called for an ‘alternative means of evaluating dental cements’. A PubMed literature search (1990-present) for “glass ionomer” highlighted 5040 manuscripts had been published in the subject area. Utilising the same search engine with “glass ionomer AND strength” as the identifiable terms 1159 manuscripts were noted. A systematic analysis of the publications revealed 303 articles were not relevant or presented no original data and 538 articles focussed on bond strength testing to various substrates. The remaining articles outlined the determination of the “strength”

of glass ionomer materials using one or more experimental techniques and were subdivided as CFS (154), diametral tensile strength (DTS) (76), uniaxial three point flexure strength (TFS) (62) and biaxial flexure strength (BFS) (26). “Strength” was also determined using load to failure tests employing arbitrary geometries (27), computational methods (11) and “other” methods (25) such as Hertzian load bearing capacity, four point flexure or the punch shear test. Therefore, the most frequently published strength testing methodology for GIs remains the CFS test, despite the reservations of McCabe et al. [4], notwithstanding the concerns in the literature regarding the validity [2-3] and reliability [4] of the method. The DTS test, TFS test and BFS test were employed in a descending order of usage in the dental literature, however, an increasing tendency to report simultaneously the results of multiple strength determination methods was evident [6-8] which may further suggest a lack of confidence in the guidance provided by the current ISO standard.

It is clear that the DTS test is frequently employed as an alternative or adjunct to the CFS test and involves diametrically compressing a disc-shaped specimen between two knife-edged supports [9]. Assuming correct alignment and specimen quality, the tensile stresses generated pull the disc in a vector half normal to the diametral plane joining the two points of contact and the mathematical calculation assumes a line contact between the load applied and specimen [2]. In practice, compression plattens rather than knife-edge supports are used and intimate point contacts are replaced by an area of contact [10]. Therefore the failure mode is hindered by the stress distribution which tends towards that for distributed loading [2] since a compressive stress exists along the loaded diameter (at

right angles to the tensile stress) [11]. Large shear stresses at the contact area also complicate the failure mode further [12]. GIs are brittle materials and the validity of the DTS methodology for brittle materials was comprehensively undermined in a landmark paper in 1990 [2]. Therefore, the DTS testing of brittle materials has essentially been made redundant from the standards testing armamentarium, although it should be noted that some research groups employ DTS testing routinely despite the accepted reservations regarding the technique [2,10-13].

In TFS testing, the length of one side of a bar-shaped specimen is in contact with the point supports and is in a state of pure tension under loading which is advantageous [14] compared with the mixture of stresses generated in CFS [2,15] or DTS [2,10-13] testing. Similarly in BFS testing, a disc-shaped specimen is placed on a continuous or interrupted circular support and loaded centrally so that the maximum tensile stresses occurs at the centre of the specimen [16]. BFS testing is reported to be advantageous over uniaxial tests such as TFS as the specimens are considered to be easier to prepare, more closely match the volume of a clinical restoration [16] and a stress field equi-biaxial away from the supports is generated distant from the specimen periphery, thereby reducing the sensitivity to specimen edge defects [13].

The aim of the current study was to examine three strength tests (CFS, BFS and TFS) for GI restoratives to assess the most appropriate strength testing methodology in terms of validity and reliability. Despite its popularity, the DTS test was not considered due to the clear theoretical evidence negating its appropriateness [2,10-13]. To eliminate mixing

[17-21] induced variability on the data sets generated encapsulated GI restoratives were employed.

Accepted Manuscript

## Materials and Methods

The three encapsulated posterior GI restoratives tested were Chemfil Rock (Dentsply DeTrey, Konstanz, Germany; LOT 1009004048, shade A3), Fuji IX<sub>GP</sub> Fast Capsule (GC Europe, Leuven, Belgium; LOT 1011181, shade A2) and Ionofil Molar AC (Voco GmbH, Cuxhaven, Germany; LOT 1105302, shade A3). Cylindrical (6.0±0.1 mm height, 4.0±0.1 mm diameter), bar-shaped (25.0±0.1 mm length, 2.0±0.1 mm height, 2.0±0.1 mm width) and disc-shaped (13.0±0.1 mm diameter, 1.0±0.1 mm thickness) specimens were prepared for CFS, TFS and BFS testing, respectively by a single experienced operator in a temperature (21±1°C) and humidity (50±5%) controlled laboratory.

Forty specimens were prepared for each of nine groups (three encapsulated GI restoratives × three strength tests). The groups of 40 were divided into eight batches of five specimens. To randomise the manufacture of the specimen batches, each batch, GI restorative and strength test was assigned a number (from 1 to 72). Prior to manufacture a number was randomly drawn, the corresponding batch prepared, with the day and time of manufacture recorded. The procedure was repeated until all batches were manufactured. To prevent operator fatigue influencing the results, six batches were manufactured daily, three in the morning and three in the afternoon. The three morning groups were prepared at 10:00±0:30, 11:00±0:30 and 12:00±0:30 while the afternoon groups were prepared at 14:00±0:30, 15:00±0:30 and 16:00±0:30.

Each GI restorative capsule was tumbled for 5 s prior to activation to aerate the powder



inside the capsule. The capsule was activated in accordance with manufacturers' instructions for 2 s to rupture the membrane separating the powder and liquid constituents [22-24]. Following activation, the capsule was placed into the holder of a Capmix (3M ESPE, Seefeld, Germany) mechanical mixing device. The Chemfil Rock capsules were vibratory mixed for 15 s [22] and the Fuji IX<sub>GP</sub> Fast Capsule and Ionofil Molar AC capsules were mixed for 10 s [23-24]. The capsules were then placed into the appropriate applicator to extrude the GI restorative into the appropriate mould.

Each CFS cylindrical (volume: 75.41 mm<sup>3</sup>) and TFS bar-shaped (volume: 100 mm<sup>3</sup>) specimen were manufactured from a single capsule. However, each BFS disc (volume: 132.75 mm<sup>3</sup>) required two capsules such that on mixing the first capsule, the second capsule was tumbled, activated and mechanically mixed to ensure consistency in specimen manufacture.

#### CFS testing

A polytetrafluoroethylene (PTFE) split-mould (capable of holding eight specimens) was used to manufacture the cylindrical specimens for CFS testing [25-26]. The PTFE base was covered with an acetate strip and the split-mould was placed on top. Alignment of the split-mould was achieved using a locating pin while nylon wedges ensured equal pressure was applied along the length of the split-mould [25-26]. The nozzle of the mixed GI restorative capsule was inserted into an unfilled cylindrical hole and the GI restorative extruded slowly to minimise air entrapment [19,27]. A second acetate strip was placed on top of the filled mould and a 1 kg glass slab was applied to isolate the specimen from the

surrounding environment. The procedure was repeated until one batch ( $n=5$ ) was made. A clamp was applied to the split-mould assembly to ensure that equal pressure was applied to all five specimens [25-26] and the assembly was transferred to a water-bath at  $37\pm 1^\circ\text{C}$  for 1 h. The split-mould was then disassembled, the individual specimens were removed, numbered from 1 to 5 (in the order of manufacture) and stored in 50 mL of distilled water at  $37\pm 1^\circ\text{C}$  for 23 h prior to CFS testing.

The diameter of each cylindrical specimen was determined at three points using a digital micrometer screw gauge accurate to  $1\ \mu\text{m}$  (Mitutoyo, Kawasaki, Japan) and the mean diameter calculated prior to CFS testing. A piece of wet filter paper (Whatman No. 1, Whatman International Ltd., Maidstone, England) was placed on the flat ends of each specimen prior to testing. A compressive load was applied to the long axis of each specimen at a loading rate of 1 mm/min (Instron Model 5565, High Wycombe, England) and the load at fracture was recorded. The CFS was determined using Eq. 1 [1]

$$\text{CFS} = \frac{4P}{\pi d^2} \quad \text{Eq 1}$$

where  $P$  was the load at fracture (N) and  $d$  was the mean specimen diameter (mm).

#### TFS testing

The bar-shaped specimens were manufactured using open-ended, knife-edged, PTFE moulds. The mixed GI restorative capsule nozzle was placed into the unfilled mould and extruded slowly whilst moving the nozzle along the length of the mould. An acetate strip was placed on top of the filled mould and a 1 kg glass slab applied to isolate the specimen and ensure consistent and reproducible specimen packing. The mould assembly was

clamped and transferred to a water-bath at  $37\pm 1^\circ\text{C}$ . The procedure was replicated to manufacture each batch ( $n=5$ ). After 1 h the specimens were removed from the mould, numbered from 1 to 5 in the order of manufacture and stored in 50 mL of distilled water at  $37\pm 1^\circ\text{C}$  for a further 23 h.

The bar-shaped specimens were placed centrally on point supports and tested at a crosshead speed of 1 mm/min using the universal testing machine. The load at fracture was recorded and the TFS was calculated using Eq. 2 [28]

$$\text{TFS} = \frac{3PL}{2bh^2} \quad \text{Eq 2}$$

where  $L$  was the span between the two supports (20 mm),  $b$  was the mean specimen width (mm), and  $h$  was the mean specimen thickness (mm). Following testing the width and thickness of each fractured fragment were measured using the micrometer screw gauge.

### BFS

The disc-shaped specimens were prepared using PTFE ring-moulds placed on a polished glass slab covered with an acetate strip. The GI restorative mix from the first capsule was extruded directly into the centre of the mould before the mix from the second capsule was added. The GI was covered with a second acetate strip and a 1 kg glass slab placed on top to spread the mix evenly throughout the mould. To ensure disc-flatness the ring-mould was secured between glass slabs using a clamp and the clamped mould assembly immersed in the water-bath at  $37\pm 1^\circ\text{C}$ . The procedure was repeated five times to manufacture one batch. The specimens were removed from the ring-moulds after 1 h,

numbered in the order of manufacture (from 1 to 5) and stored in 50 mL of distilled water at  $37\pm 1^\circ\text{C}$  for a further 23 h.

The disc-shaped specimens were placed on a 10 mm diameter knife-edge ring annulus covered with a thin sheet of rubber (to facilitate uniform loading) [29] and centrally loaded with a 2 mm ball-indenter using the universal testing machine at a crosshead speed of 1 mm/min. The load at fracture ( $P$ ) was recorded and the BFS calculated using Eq. 3 [30].

$$\text{BFS} = \frac{P}{h^2} \left\{ (1 + \nu) \left[ 0.485 \ln \left( \frac{a}{h} \right) + 0.52 \right] + 0.48 \right\} \quad \text{Eq 3}$$

where  $a$  was the radius of the knife-edge support (10 mm) and  $\nu$  was Poisson's ratio (0.3 for GI restoratives [31]). The thickness of each fractured fragment was measured at the point of fracture using the micrometer and the mean thickness  $h$  was determined.

### Statistical analysis

The CFS, TFS and BFS data were statistically analysed individually at a significance value of  $p=0.05$  using software (SPSS 12.0.1; SPSS Inc., Chicago, IL, USA). The data was initially explored using Shapiro-Wilk methods to test for normality. Thirty six regression analyses were conducted (four for each individual group (GI restorative  $\times$  strength test)) to check if the data sets were significantly influenced by each of four factors (batch, specimen number (in the order of manufacture), day and time of manufacture. Three one-way analyses of variance (ANOVAs), one each for CFS, TFS and BFS, were conducted to compare between GI restorative group means and Tukey's Post-hoc tests were used where appropriate. The reliability of the three strength tests was

assessed by determining the coefficient of variation (CoV), namely the ratio of the standard deviation to the mean of each batch tested. CoV is a useful measure of reliability where the means generated by the three different tests for each material are expected to widely differ (in the current study as a consequence of the dissimilar stress fields and volumes under stress). To test for differences between the reliability of the three tests, the CoV values for each test (n=24: eight batches × three GI restoratives) were pooled and a one-way ANOVA was conducted.

Accepted Manuscript

## Results

For the three encapsulated GI restoratives investigated, the CFS, TFS and BFS batch means and overall group means (with associated standard deviations and CoVs) are shown in Tables 1-3, respectively. The variances of the strength data for each group under investigation were checked using Levene's test of homogeneity and all groups were homogeneous ( $p>0.05$ ). The frequency distributions of the CFS, TFS and BFS data with superimposed normal curves for the three GI restoratives are shown in Figures 1-3, respectively. Additionally, the Shapiro-Wilk test identified that all groups were normally distributed ( $p>0.074$ ).

The regression analyses showed the CFS data for the three GI restoratives were not significantly influenced by batch ( $p>0.058$ ), specimen number ( $p>0.210$ ), day ( $p>0.068$ ) or time of manufacture ( $p>0.120$ ) (Table 4). There was also no significant effect of batch ( $p>0.349$ ), specimen number ( $p>0.255$ ), day ( $p>0.383$ ), or time of manufacture ( $p>0.491$ ) on the TFS data for the three GI restorative materials (Table 5). Similarly, batch ( $p>0.126$ ), specimen number ( $p>0.259$ ), day ( $p>0.080$ ) or time of manufacture ( $p>0.090$ ) had no significant influence on the BFS data for the three GI restorative materials (Table 6).

The one-way ANOVA for the CFS data showed a significant difference between GI restoratives ( $p=0.001$ ) and the Tukey's Post-hoc tests highlighted the CFS of Ionofil Molar AC was significantly reduced compared with Fuji IX<sub>GP</sub> Fast Capsule ( $p=0.001$ ) but

not Chemfil Rock ( $p=0.138$ ). The one-way ANOVAs showed no significant differences between the TFS ( $p=0.271$ ) and BFS ( $p=0.134$ ) data for the GI restoratives. The pooled CoV values for the CFS, TFS and BFS tests showed a mean of 0.104, 0.109 and 0.116, respectively (Table 1). Pooling all the CoV values and conducting a one-way ANOVA showed no significant difference between the CFS, TFS and BFS tests ( $p=0.632$ ) (Table 7).

Accepted Manuscript

## Discussion

In order to experimentally objectively assess the currently recommended and alternative strength testing methodologies for GI materials it was essential to generate discrete data-sets free from bias and confounding factors. In Part 1, the authors highlighted the importance of rigorously controlling operator and environmental variables during the manufacture of CFS test specimens and subsequent testing [5]. In doing so it was demonstrated that with a realistic sample size ( $n=30$ ) the ‘test-house variability’ observed by McCabe et al. [4] for CFS testing could be eliminated. In the current investigation by using regression analyses it was demonstrated that the CFS, TFS and BFS data were not significantly influenced by when (day or time of day) or in what sequence (batch or specimen order) the specimens were manufactured. Therefore simple randomisation of the manufactured specimens would not have been appropriate in the context of the current investigation as different geometries were manufactured from three GI restoratives. The authors suggest that similar initial analyses should form a routine part of experimental data exploration prior to the selection of statistical tools to test for differences between the experimental variables.

As expected the CFS value was the highest “strength” value generated using the three testing methodologies since the load bearing capacity of brittle materials such as GI restoratives is greater in compression than in tension [32]. In contrast to failure originating in a pure tensile stress field, which is usually localised to a narrow area of damage, compressive loading results in a much larger damage zone. Under compressive



loading the specimen expands laterally resulting in the formation of splitting ‘wing’ cracks originating from the primary flaws which are inherent to the material or introduced during mixing or specimen preparation [33]. The cracks propagate parallel to the compression axis as the lateral deformation of the specimen increases during test progression. However, the mechanism leading to ultimate failure under compressive loading remains considerably more complex than that observed in a tensile strength test. The formation of ‘wing’ cracks is also associated with secondary crack propagation and unpredictable crack linkage which coupled with the potential for bending induced failure of peripheral columns split from the specimen during the progress of the test [33] results in the specimen collapsing due to ‘some unresolved combination of tension and shear’ [2]. The stress at failure is subsequently calculated from pre-test specimen geometry and load at failure and does not take account of the actual failure mechanism [2].

The TFS testing methodology imposes a compressive stress on the top surface of the specimen. The specimen fails in a reproducible manner from the tensile stresses generated in the bottom surface which is in contact with the point supports [13]. Therefore the stress is not uniform, varying to a maximum tensile stress at the bottom surface which accentuates the effect of surface finishing condition [14]. Surface flaws introduced by the operator during mixing or specimen manufacture can be eliminated by polishing thereby improving the TFS [29]. In the dental literature TFS testing has been employed frequently for dental ceramics and resin-based composite (RBC) materials. The preparation of dental ceramic bar-shaped specimens is difficult because of problems associated with condensing slurry consistencies into the mould without introducing air

bubbles, notwithstanding the impact of surface finishing condition which can markedly influence the TFS results achieved [14]. For RBC materials, the efficiency of the overlapping irradiation regime used in the manufacture of bar-shaped specimens has been questioned [34] in terms of uncontrolled initiation on polymerisation [35], non-homogeneously cured specimens [36] and inconsistent polymerisation along the length of the specimen [37-38] which further complicates the choice of testing methodology. In the current study, the bar-shaped specimens were easier to consolidate into the mould owing to the nozzle of the encapsulated GI capsules manifest as the low CoV of the pooled TFS data of 0.109. However, it is anticipated that the reliability of specimen manufacture from hand-mixed GI restoratives would be markedly increased compared with the encapsulated products owing to mixing induced variability [19] and consolidation issues on mould filling [20].

Testing in biaxial flexure can be performed using a variety of testing assemblies (ball-, ring- or piston-on-ring and ball-, ring- or piston-on-three-ball) with the ball-on-ring loading configuration advocated by de With and Wagemans [39] as being the most reliable although Williams et al. [40] reported the type of support (ring or three-ball) was not important and did not influence the result. Further studies showed no significant impact of the thin sheet of rubber in facilitating uniform loading [29]. However, a significant advantage of the BFS test is the ease of specimen preparation due to the controllable specimen geometry [41] which contributed to the low CoV of the pooled BFS data of 0.116. It is suggested that not only could encapsulated GI restorative specimens be made reliably but specimen consolidation is simplified for the controllable

mould geometry. BFS disc-shaped specimens are extensively used in dental ceramic [41] and RBC [37] research for the reasons of controllable specimen geometry.

In the current study, the CoV of the pooled CFS data did not significantly differ from BFS or TFS and hence provided no advantage or disadvantage in terms of the reliability of the data. However, when the original strength data for the three strength tests were analysed using one-way ANOVAs significant differences were evident in the CFS for the three GIs investigated but no significant differences between materials were shown for the TFS and BFS data. The observation is concerning given that the failure modes (and origin of failure) for specimens in TFS and BFS testing are likely to be consistently reproducible. The authors propose that such findings may not necessarily reflect differences between the “strength” of the different GI restoratives but may arise due to greater sensitivity to material dependent differences during specimen fabrication. It has been demonstrated that the operator technique when filling a CFS mould with a particular encapsulated GI restorative significantly impacted upon the recorded CFS data [20-21] by varying the pore inclusion distribution. These factors were further compounded when filling the mould with a hand-mixed GI restorative [17]. The CFS test has been demonstrated through the literature to be an invalid measure of “strength” and offers no advantages in the context of the strength data it generates when compared with TFS or BFS testing. Consequently, the authors propose that the CFS test should be replaced by the International Organisation for Standardisation and should no longer be advocated for the predictive performance modelling of GI restoratives. Both TFS and BFS tests are valid strength measures and the current study could not demonstrate statistically one test

to be more advantageous than the other. However, the authors suggest that specimen fabrication for BFS testing is simpler and less likely to be sensitive to inter-operator variability.

### **Conclusion**

The CFS test specified for GIs in the International Organisation for Standardisation (ISO 9917-1: 2003) should be replaced and should no longer be advocated for the predictive performance modelling of GI restoratives.

## References

1. International Organization for Standardization. ISO 9917-1 – Dentistry – water-based cements Part1: Powder/liquid acid-base cements. 1st ed. 2003.
2. Darvell BW. Uniaxial compression tests and the validity of indirect tensile strength. *Journal of Materials Science* 1990;**25**:757-80.
3. Wilson AD. Dental cements – general. Zinc oxide dental cements. Dental cement based on ion-leachable glasses In: Von Fraunhofer JA, editor. Scientific Aspects of Dental Materials. London: Butterworths, 1975. p 131-221.
4. McCabe JF, Watts DC, Wilson HJ, Worthington HV. An investigation of test-house variability in the mechanical testing of dental materials. *Journal of Dentistry* 1990;**18**:90-7.
5. Fleming GJP, Dowling AH, Addison O. The crushing truth about glass ionomer restoratives: exposing the standard of the standard. *Journal of Dentistry* 2011 (under review).
6. Moshaverinia A, Ansari S, Movasaghi Z, Billington RW, Darr JA, Rehman IU. Modification of conventional glass-ionomer cements with N-vinylpyrrolidone containing polyacids, nano-hydroxy and fluoroapatite to improve mechanical properties. *Dental Materials* 2008;**24**:1381-90.
7. Moshaverinia A, Ansari S, Moshaverinia M, Roohpour N, Darr JA, Rehman I. Effects of incorporation of hydroxyapatite and fluoroapatite nanobioceramics into conventional glass ionomer cements (GIC). *Acta Biomaterialia* 2008;**4**:432-40.

8. Bresciani E, Barata Tde J, Fagundes TC, Adachi A, Terrin MM, Navarro MF. Compressive and diametral tensile strength of glass ionomer cements. *Journal of Applied Oral Sciences* 2004;**12**:344-8.
9. Wright PJF. Comments on an indirect tensile test on concrete cylinders. *Magazine of Concrete Research* 1955;**1**:87-96.
10. Peltier R. Theoretical investigation of the Brazilian test. *Union Testing of Research Laboratory Materials Structures* 1954;**19**:29-69.
11. Kendall K. Complexities of compression failure. *Proceedings of the Royal Society of London* 1978;**A.361**:245-63.
12. Lloyd CH, Mitchell L. The fracture toughness of tooth coloured restorative materials. *Journal of Oral Rehabilitation* 1984;**11**:257-72.
13. Berenbaum R, Brodie I. Measurement of tensile strength of brittle materials. *British Journal of Applied Physics* 1959;**10**:281-7.
14. Giovan MN, Sines G. Biaxial and uniaxial data for statistical comparison of a ceramic's strength. *Journal of the American Ceramic Society* 1979;**62**:510-5.
15. Fairhurst CW. Compressive properties of dental gypsum. *Journal of Dental Research* 1960;**39**:812-24.
16. Ban S, Hasegawa J, Anusavice KJ. Effect of loading conditions on bi-axial flexure strength of dental cements. *Dental Materials* 1992;**8**:100-4.
17. Fleming GJP, Farooq AA, Barralett JE. The influence of powder/liquid mixing ratio on the performance of a range of glass-ionomer cements. *Biomaterials* 2003;**24**:4173-9.

18. Billington RW, Williams JA, Pearson GJ. Variation in powder/liquid ratio of a restorative glass-ionomer cement used in dental practice. *British Dental Journal* 1990;**169**:164-7.
19. Dowling AH, Fleming GJP. Is encapsulation of posterior glass-ionomer restoratives the solution to clinically induced variability introduced on mixing? *Dental Materials* 2008;**24**:957-66.
20. Fleming GJP, Kenny SM, Barralet JE. The optimisation of the initial viscosity of an encapsulated glass-ionomer filling material following different mechanical mixing regimes. *Journal of Dentistry* 2006;**34**:155-63.
21. Fleming GJP, Zala DM. An assessment of encapsulated versus hand-mixed glass ionomer restoratives. *Operative Dentistry* 2003;**28**:168-77.
22. Product specification for ChemFlex Rock (Dentsply DeTrey, Konstanz, Germany).
23. Product specification for Fuji IX<sub>GP</sub> Fast Capsule (GC Europe, Leuven, Belgium).
24. Product specification for Ionofil Molar AC (Voco GmbH, Cuxhaven, Germany).
25. Fleming GJP, Marquis PM, Shortall ACC. The influence of clinically induced variability on the distribution of compressive fracture strengths of a hand-mixed zinc phosphate dental cement. *Dental Materials* 1999;**15**:87-97.
26. Fleming GJP, Shelton RM, Landini G, Marquis PM. Encapsulated versus hand-mixed zinc phosphate dental cement. *Biomaterials* 1999;**20**:2147-53.
27. Dowling AH, Fleming GJP. Are encapsulated anterior glass-ionomer restoratives better than their hand-mixed equivalents? *Journal of Dentistry* 2009;**37**:133-40.
28. International Organization for Standardization. ISO 4049 - Dentistry - polymer-based filling, restorative and luting materials. 3rd ed. 2000: p.15-18.

29. Ban S, Anusavice KJ. Influence of test method on failure stress of brittle materials. *Journal of Dental Research* 1990;**69**:1791-9.
30. Timoshenko S, Woinowsky-Krieger S. Symmetrical bending of circular plates. In *Theory of Plates and Shells*. 2nd ed. New York: McGraw-Hill 1959 p. 87-121.
31. Akinmade AO, Nicholson JW. Poisson's ratio of glass-polyalkenoate ("glass-ionomer") cements determined by an ultrasonic pulse method. *Journal of Materials Science: Materials in Medicine* 1995;**6**:483-5.
32. Ritter JE. Critique of test methods for lifetime predictions. *Dental Materials* 1995;**11**:147-51.
33. Renshaw CE, Schulson EM. Universal behaviour in compressive failure of brittle materials. *Nature* 2001;**412**:897-900.
34. Bhamra GS, Fleming GJP. Effects of halogen light irradiation variables (tip diameter, irradiance, irradiation protocol) on flexural strength properties of resin-based composites. *Journal of Dentistry* 2008;**36**:643-50
35. Mehl A, Hickel R, Kunzelmann K-H. Physical properties and gap formation of light-cured composites with and without 'soft-start polymerization'. *Journal of Dentistry* 1997;**25**:321-30.
36. Ferracane JL, Ferracane LL, Musanje L. Effect of light activation method on flexural properties of dental composites. *American Journal of Dentistry* 2003;**16**:318-22.
37. Palin WM, Fleming GJP, Burke FJT, Marquis PM, Randall RC. The reliability in flexural strength testing of a novel dental composite. *Journal of Dentistry* 2003;**31**:549-57.



38. Palin WM, Fleming GJP, Marquis PM. The reliability of standardized flexure strength testing procedures for a light-activated resin-based composite. *Dental Materials* 2005;**21**:911-9.
39. de With G, Wagemans HHM. Ball-on-ring test revisited. *Journal of the American Ceramic Society* 1989;**72**:1538-41.
40. Williams JA, Billington RW, Pearson GJ. The effect of the disc support system on biaxial tensile strength of a glass ionomer cement. *Dental Materials* 2002;**18**:376-9.
41. Fleming GJP, Shaini FJ, Marquis PM. An assessment of the influence of mixing induced variability on the bi-axial flexure strength of dentine porcelain discs and the implications for laboratory testing of porcelain specimens. *Dental Materials* 2000;**16**:114-9.

Tables:

Specimen Number	Chemfil Rock	Fuji IX <sub>GP</sub> Fast Capsule	Ionofil Molar AC
1	120.76	138.57	115.94
2	141.45	156.11	112.36
3	131.45	115.44	117.81
4	134.27	129.32	113.31
5	117.48	133.89	118.93
<b>Mean±SD</b>	129.08±9.87	134.67±14.78	115.67±2.82
<b>CoV</b>	0.076	0.110	0.024
6	125.51	127.49	113.70
7	147.72	138.60	118.76
8	122.86	184.41	121.13
9	125.62	135.33	143.63
10	113.37	110.72	120.30
<b>Mean±SD</b>	127.02±12.61	139.31±27.42	123.50±11.62
<b>CoV</b>	0.099	0.197	0.094
11	116.75	119.33	110.31
12	108.21	127.08	104.45
13	144.89	125.50	129.96
14	136.59	150.14	99.70
15	157.15	139.81	111.35
<b>Mean±SD</b>	132.72±20.10	132.37±12.41	111.15±11.52
<b>CoV</b>	0.151	0.094	0.104
16	122.45	131.53	154.73
17	117.62	128.01	153.62
18	122.22	134.07	132.60
19	138.74	139.80	126.33
20	113.95	137.25	126.93
<b>Mean±SD</b>	123.00±9.48	134.13±4.64	138.84±14.21
<b>CoV</b>	0.077	0.035	0.102
21	131.72	155.92	133.75
22	130.65	146.21	103.94
23	107.43	127.83	102.76
24	118.96	118.80	124.63
25	147.59	147.38	137.49
<b>Mean±SD</b>	127.27±15.06	139.23±15.34	120.51±16.36
<b>CoV</b>	0.118	0.110	0.136
26	146.76	155.32	109.62
27	141.32	160.25	118.35
28	146.54	134.14	105.52
29	130.96	122.96	116.39
30	160.22	133.48	128.13
<b>Mean±SD</b>	145.16±10.58	141.23±15.85	115.60±8.70

<b>CoV</b>	0.073	0.112	0.075
<b>31</b>	133.72	134.12	125.24
<b>32</b>	119.54	160.52	138.91
<b>33</b>	149.28	131.65	108.80
<b>34</b>	126.95	109.95	129.15
<b>35</b>	121.13	126.83	146.68
<b>Mean±SD</b>	130.12±12.07	132.62±18.23	129.76±14.40
<b>CoV</b>	0.093	0.137	0.111
<b>36</b>	113.68	125.03	142.19
<b>37</b>	129.56	141.64	127.99
<b>38</b>	115.08	138.42	141.33
<b>39</b>	145.62	170.75	143.05
<b>40</b>	126.09	105.67	117.30
<b>Mean±SD</b>	126.00±12.93	136.30±23.90	134.37±11.37
<b>CoV</b>	0.103	0.175	0.085
<b>Total Mean±SD</b>	130.05±13.59	136.23±16.44	123.68±14.34
<b>Mean CoV</b>	0.099	0.121	0.091

Table 1: The mean compressive fracture strength (CFS) ± standard deviation (SD) and coefficient of variation (CoV) for the three encapsulated GI restoratives.

Specimen Number	Chemfil Rock	Fuji IX <sub>GP</sub> Fast Capsule	Ionofil Molar AC
1	34.19	26.64	33.80
2	33.87	36.35	40.09
3	38.62	39.00	40.73
4	38.22	33.11	44.62
5	40.81	34.16	33.38
<b>Mean±SD</b>	<b>37.14±3.01</b>	<b>33.85±4.62</b>	<b>38.52±4.83</b>
<b>CoV</b>	<b>0.081</b>	<b>0.136</b>	<b>0.125</b>
6	38.71	41.36	36.71
7	42.87	36.94	37.50
8	38.08	43.50	40.03
9	35.70	33.56	43.91
10	30.77	44.47	40.85
<b>Mean±SD</b>	<b>37.23±4.44</b>	<b>39.97±4.61</b>	<b>39.80±2.87</b>
<b>CoV</b>	<b>0.119</b>	<b>0.115</b>	<b>0.072</b>
11	43.72	44.87	43.09
12	32.49	46.91	46.04
13	34.58	32.97	48.63
14	45.91	35.37	39.95
15	33.01	28.36	36.76
<b>Mean±SD</b>	<b>37.94±6.37</b>	<b>37.70±7.92</b>	<b>42.89±4.72</b>
<b>CoV</b>	<b>0.168</b>	<b>0.210</b>	<b>0.110</b>
16	34.44	30.11	39.37
17	40.59	37.76	36.80
18	40.97	38.78	35.77
19	42.61	36.80	38.86
20	41.76	28.70	38.56
<b>Mean±SD</b>	<b>40.07±3.25</b>	<b>34.43±4.67</b>	<b>37.87±1.52</b>
<b>CoV</b>	<b>0.081</b>	<b>0.136</b>	<b>0.040</b>
21	36.75	36.19	34.76
22	41.66	39.78	45.73
23	40.34	43.59	38.59
24	39.11	38.20	36.75
25	44.65	41.85	45.22
<b>Mean±SD</b>	<b>40.50±2.94</b>	<b>39.92±2.92</b>	<b>40.21±5.00</b>
<b>CoV</b>	<b>0.073</b>	<b>0.073</b>	<b>0.124</b>
26	34.03	37.25	35.66
27	35.42	35.53	48.00
28	34.44	26.95	33.74
29	45.62	36.16	30.66
30	31.16	38.95	48.69
<b>Mean±SD</b>	<b>36.13±5.54</b>	<b>34.97±4.67</b>	<b>39.35±8.40</b>

<b>CoV</b>	0.153	0.133	0.214
<b>31</b>	35.23	41.86	43.34
<b>32</b>	39.61	28.51	47.27
<b>33</b>	41.08	40.67	42.11
<b>34</b>	43.74	32.87	32.88
<b>35</b>	36.99	38.67	51.25
<b>Mean±SD</b>	39.33±3.35	36.52±5.65	43.37±6.87
<b>CoV</b>	0.085	0.155	0.158
<b>36</b>	32.59	37.07	34.91
<b>37</b>	35.98	33.49	36.36
<b>38</b>	42.14	39.21	35.41
<b>39</b>	41.21	38.44	35.59
<b>40</b>	36.97	30.80	34.39
<b>Mean±SD</b>	37.78±3.92	35.80±3.55	35.33±0.74
<b>CoV</b>	0.104	0.099	0.021
<b>Total Mean±SD</b>	38.27±4.13	36.64±5.07	39.67±5.16
<b>Mean CoV</b>	0.108	0.132	0.108

Table 2: The mean uniaxial three point flexure strength (TFS) ± standard deviation (SD) and coefficient of variation (CoV) for the three encapsulated GI restoratives.

Specimen Number	Chemfil Rock	Fuji IX <sub>GP</sub> Fast Capsule	Ionofil Molar AC
1	68.70	66.85	85.37
2	73.37	55.61	65.57
3	74.34	62.84	59.38
4	89.51	85.75	72.52
5	63.05	76.23	77.31
<b>Mean±SD</b>	<b>73.79±9.86</b>	<b>69.46±11.77</b>	<b>72.03±10.10</b>
<b>CoV</b>	<b>0.134</b>	<b>0.169</b>	<b>0.140</b>
6	69.96	84.19	52.02
7	70.95	66.56	76.19
8	66.15	67.79	47.94
9	74.63	56.74	72.41
10	79.98	63.81	66.29
<b>Mean±SD</b>	<b>72.33±5.23</b>	<b>67.82±10.11</b>	<b>62.97±12.46</b>
<b>CoV</b>	<b>0.072</b>	<b>0.149</b>	<b>0.198</b>
11	76.91	77.54	70.53
12	52.86	83.32	71.04
13	63.19	77.16	71.78
14	82.02	82.89	64.30
15	63.10	64.79	75.93
<b>Mean±SD</b>	<b>67.62±11.74</b>	<b>77.14±7.48</b>	<b>70.72±4.17</b>
<b>CoV</b>	<b>0.174</b>	<b>0.097</b>	<b>0.059</b>
16	59.57	79.25	78.44
17	71.24	65.86	86.37
18	85.58	70.71	86.00
19	65.14	63.87	63.29
20	64.23	66.09	78.66
<b>Mean±SD</b>	<b>69.15±10.08</b>	<b>69.16±6.18</b>	<b>78.55±9.35</b>
<b>CoV</b>	<b>0.146</b>	<b>0.089</b>	<b>0.119</b>
21	67.59	64.09	64.61
22	72.04	70.14	57.61
23	76.88	71.09	82.81
24	66.22	80.88	79.26
25	79.16	68.51	57.66
<b>Mean±SD</b>	<b>72.38±5.64</b>	<b>70.94±6.17</b>	<b>68.39±11.96</b>
<b>CoV</b>	<b>0.078</b>	<b>0.087</b>	<b>0.175</b>
26	68.31	75.34	67.94
27	71.87	71.97	79.37
28	83.77	59.38	65.21
29	63.16	72.52	65.62
30	79.40	76.91	68.54
<b>Mean±SD</b>	<b>73.30±8.31</b>	<b>71.22±6.92</b>	<b>69.34±5.79</b>

<b>CoV</b>	0.113	0.097	0.084
<b>31</b>	79.17	63.33	70.36
<b>32</b>	80.86	67.64	60.43
<b>33</b>	77.28	59.99	81.77
<b>34</b>	78.19	64.89	76.01
<b>35</b>	78.12	66.95	59.71
<b>Mean±SD</b>	78.72±1.37	64.56±3.07	69.66±9.64
<b>CoV</b>	0.017	0.048	0.138
<b>36</b>	67.09	64.73	71.02
<b>37</b>	76.00	63.74	76.20
<b>38</b>	80.46	59.59	69.08
<b>39</b>	77.50	67.42	54.28
<b>40</b>	76.66	65.48	63.79
<b>Mean±SD</b>	75.54±5.02	64.19±2.90	66.87±8.32
<b>CoV</b>	0.067	0.045	0.124
<b>Total Mean±SD</b>	72.86±7.83	69.31±7.76	69.82±9.48
<b>Mean CoV</b>	0.100	0.098	0.130

Table 3: The mean biaxial flexure strength (BFS) ± standard deviation (SD) and coefficient of variation (CoV) for the three encapsulated GI restoratives.

<b>Material</b>	<b>Factor</b>	<b>Line equation</b>	<b>df</b>	<b>r<sup>2</sup></b>	<b>p</b>
<b>Chemfil Rock</b>	<b>Batch</b>	$y = 0.424x + 128.14$	38	0.005	0.657
	<b>Specimen Number</b>	$y = 1.411x + 125.81$	38	0.022	0.360
	<b>Time of Day</b>	$y = -1.509x + 134.95$	38	0.062	0.120
	<b>Day</b>	$y = 0.325x + 127.73$	38	0.007	0.621
<b>Fuji IX<sub>GP</sub> Fast Capsule</b>	<b>Batch</b>	$y = 0.115x + 125.71$	38	0.001	0.921
	<b>Specimen Number</b>	$y = -2.324x + 143.20$	38	0.041	0.210
	<b>Time of Day</b>	$y = 0.601x + 134.50$	38	0.004	0.689
	<b>Day</b>	$y = 0.038x + 135.92$	38	0.001	0.975
<b>Ionofil Molar AC</b>	<b>Batch</b>	$y = 1.872x + 115.25$	38	0.092	0.058
	<b>Specimen Number</b>	$y = 0.264x + 122.88$	38	0.001	0.872
	<b>Time of Day</b>	$y = 1.556x + 118.03$	38	0.048	0.174
	<b>Day</b>	$y = 1.374x + 113.89$	38	0.121	0.068

Table 4: Regression analysis results for the CFS data for the three GI restoratives. Key:

df: degrees of freedom, r<sup>2</sup>: the coefficient of determination and p: probability.

<b>Material</b>	<b>Factor</b>	<b>Line equation</b>	<b>df</b>	<b>r<sup>2</sup></b>	<b>p</b>
<b>Chemfil Rock</b>	<b>Batch</b>	$y = 0.119x + 37.73$	38	0.004	0.682
	<b>Specimen Number</b>	$y = 0.532x + 36.67$	38	0.034	0.255
	<b>Time of Day</b>	$y = -0.300x + 39.39$	38	0.009	0.557
	<b>Day</b>	$y = 0.071x + 37.69$	38	0.003	0.735
<b>Fuji IX<sub>GP</sub> Fast Capsule</b>	<b>Batch</b>	$y = -0.075x + 36.93$	38	0.001	0.833
	<b>Specimen Number</b>	$y = -0.369x + 37.75$	38	0.011	0.523
	<b>Time of Day</b>	$y = 0.314x + 35.47$	38	0.013	0.491
	<b>Day</b>	$y = -0.094x + 37.13$	38	0.003	0.718
<b>Ionofil Molar AC</b>	<b>Batch</b>	$y = 0.556x + 36.31$	38	0.023	0.349
	<b>Specimen Number</b>	$y = -0.462x + 40.19$	38	0.006	0.633
	<b>Time of Day</b>	$y = 0.697x + 36.71$	38	0.012	0.499
	<b>Day</b>	$y = 0.344x + 36.31$	38	0.020	0.383

Table 5: Regression analysis results for the TFS data for the three GI restoratives. Key: as

for Table 4.



Material	Factor	Line equation	df	r <sup>2</sup>	p
Chemfil Rock	Batch	$y = 0.767x + 69.40$	38	0.052	0.158
	Specimen Number	$y = 0.999x + 69.86$	38	0.033	0.259
	Time of Day	$y = 1.224x + 68.27$	38	0.074	0.090
	Day	$y = 0.478x + 69.87$	38	0.045	0.190
Fuji IX <sub>GP</sub> Fast Capsule	Batch	$y = -0.823x + 73.01$	38	0.061	0.126
	Specimen Number	$y = -0.287x + 70.17$	38	0.003	0.745
	Time of Day	$y = -1.556x + 75.73$	38	0.066	0.109
	Day	$y = -0.860x + 73.29$	38	0.078	0.080
Ionofil Molar AC	Batch	$y = 0.159x + 68.56$	38	0.001	0.826
	Specimen Number	$y = -0.353x + 70.33$	38	0.002	0.764
	Time of Day	$y = 1.175x + 65.16$	38	0.017	0.427
	Day	$y = -0.093x + 69.71$	38	0.001	0.839

Table 6: Regression analysis results for the BFS data for the three GI restoratives. Key: as for Table 4.

Source of Variation	df	SS	MS	F	p
Strength Test	2	0.002	0.001	0.461	0.632
Residual	69	0.135	0.002		
Total	72	1.003			

Table 7: One-way ANOVA for and coefficient of variation data. Key: SS: sum of squares, MS: mean square, and F: test of significance.

**Figure Captions:**

Figure 1: Frequency distributions of the CFS data for (a) Chemfil Rock, (b) Fuji IX<sub>GP</sub> Fast Capsule and (c) Ionofil Molar AC with normal distribution curves superimposed.

Figure 2: Frequency distributions of the TFS data for (a) Chemfil Rock, (b) Fuji IX<sub>GP</sub> Fast Capsule and (c) Ionofil Molar AC with normal distribution curves superimposed.

Figure 3: Frequency distributions of the BFS data for (a) Chemfil Rock, (b) Fuji IX<sub>GP</sub> Fast Capsule and (c) Ionofil Molar AC with normal distribution curves superimposed.

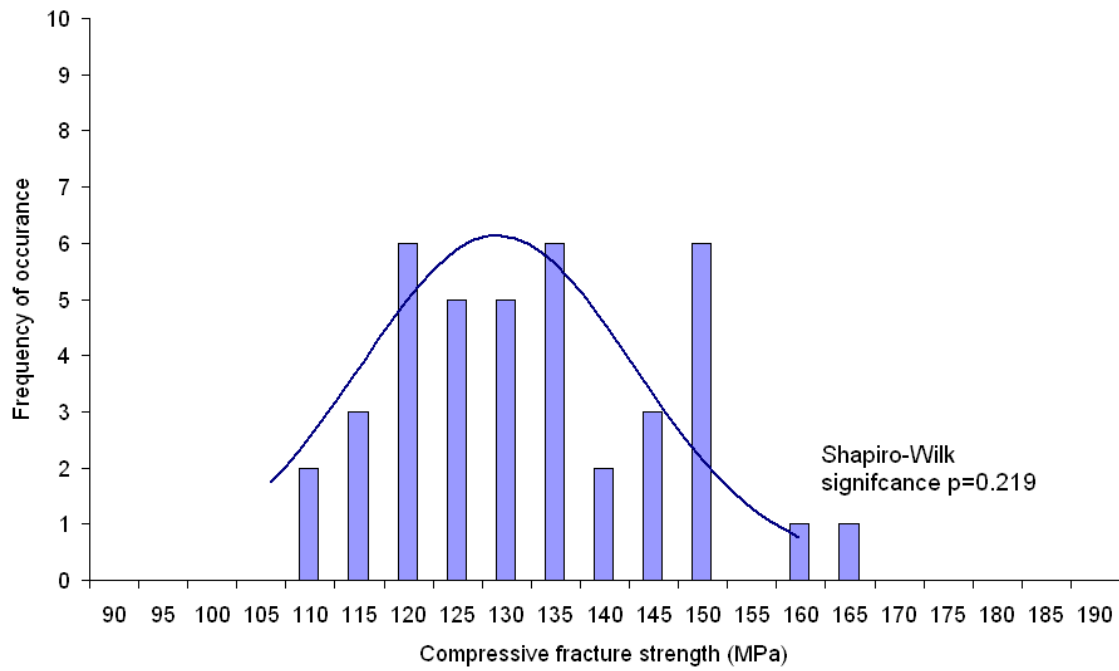


Figure 1a

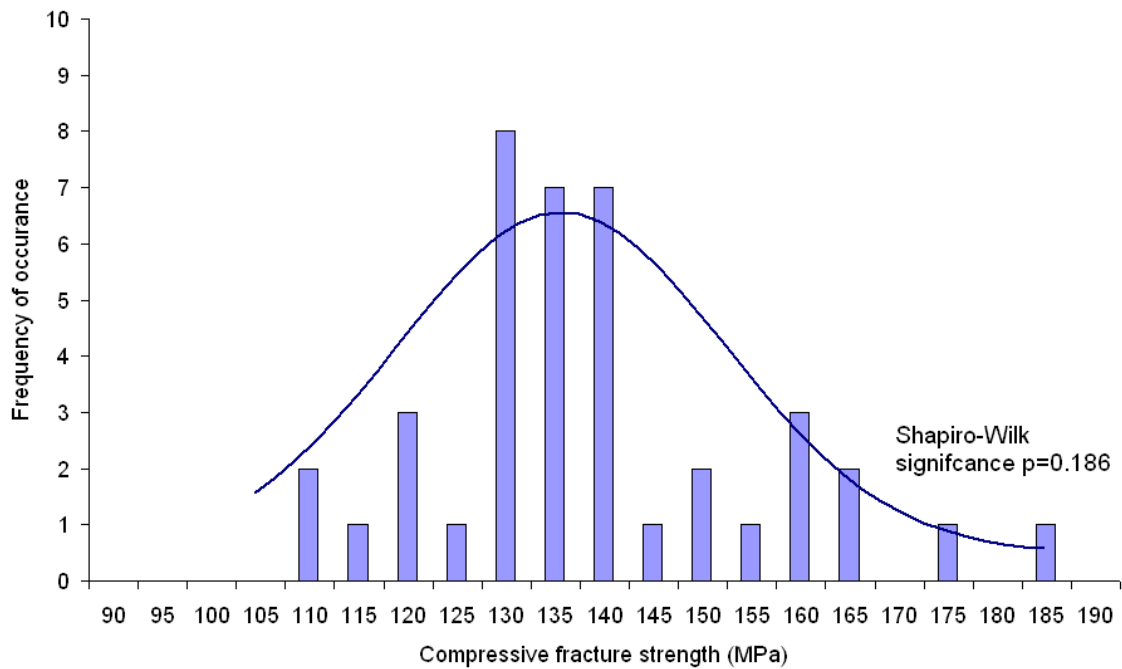


Figure 1b

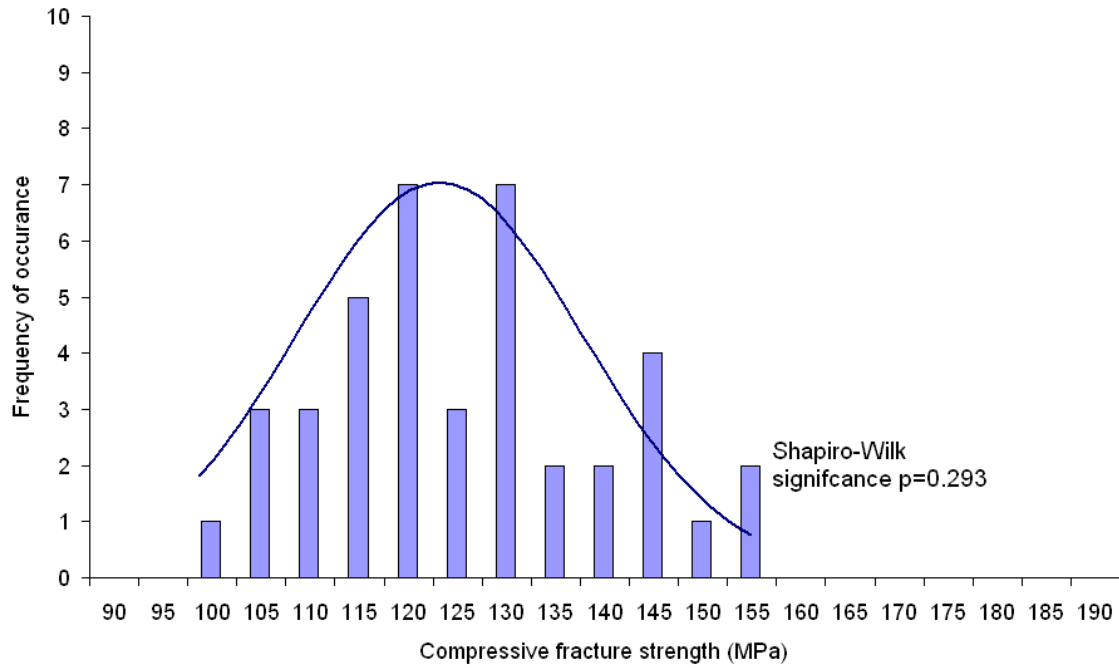


Figure 1c

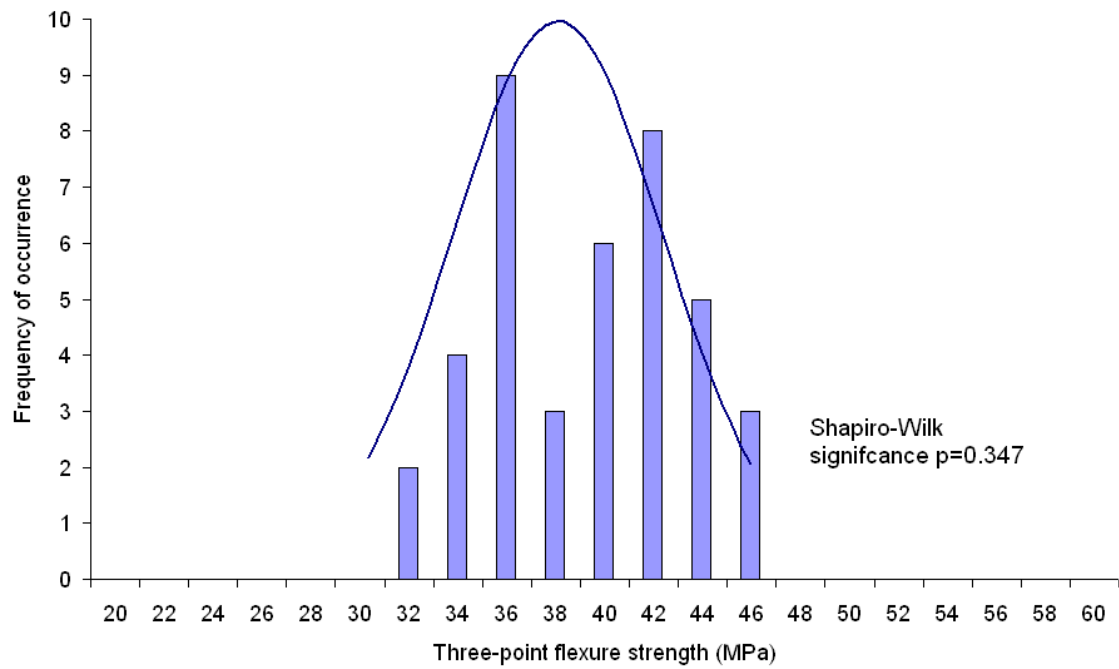


Figure 2a

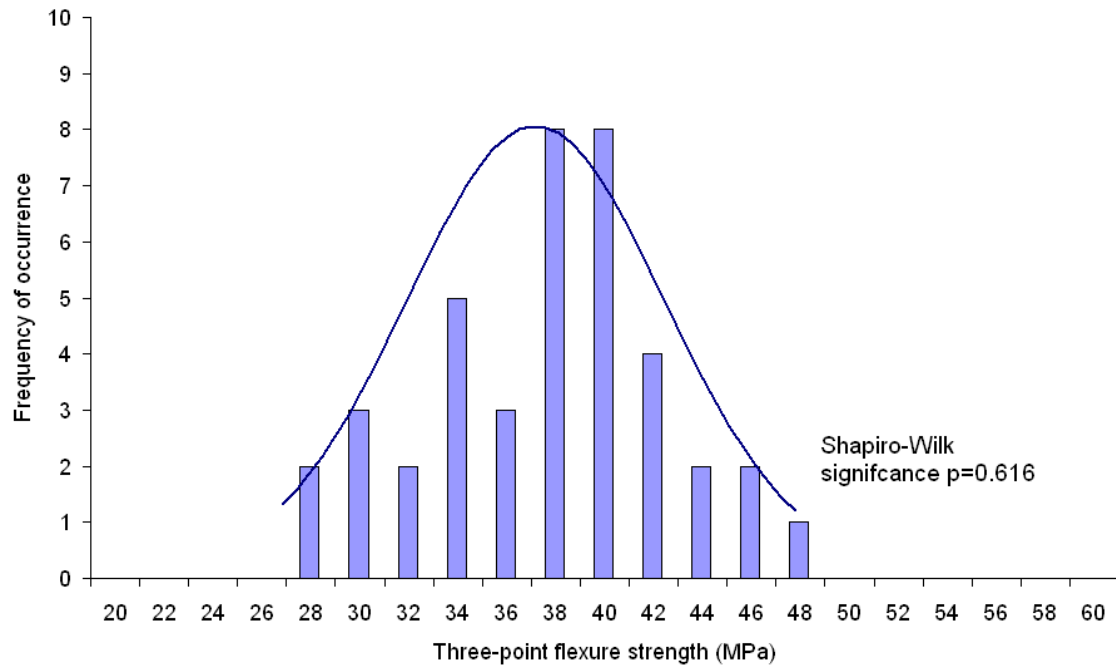


Figure 2b

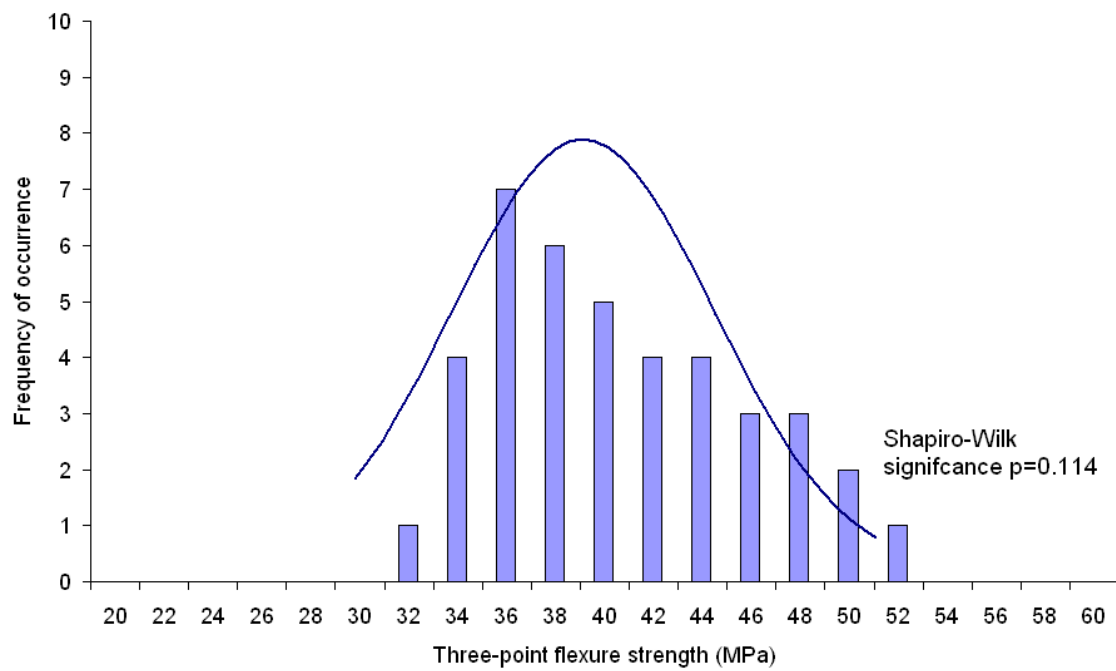


Figure 2c

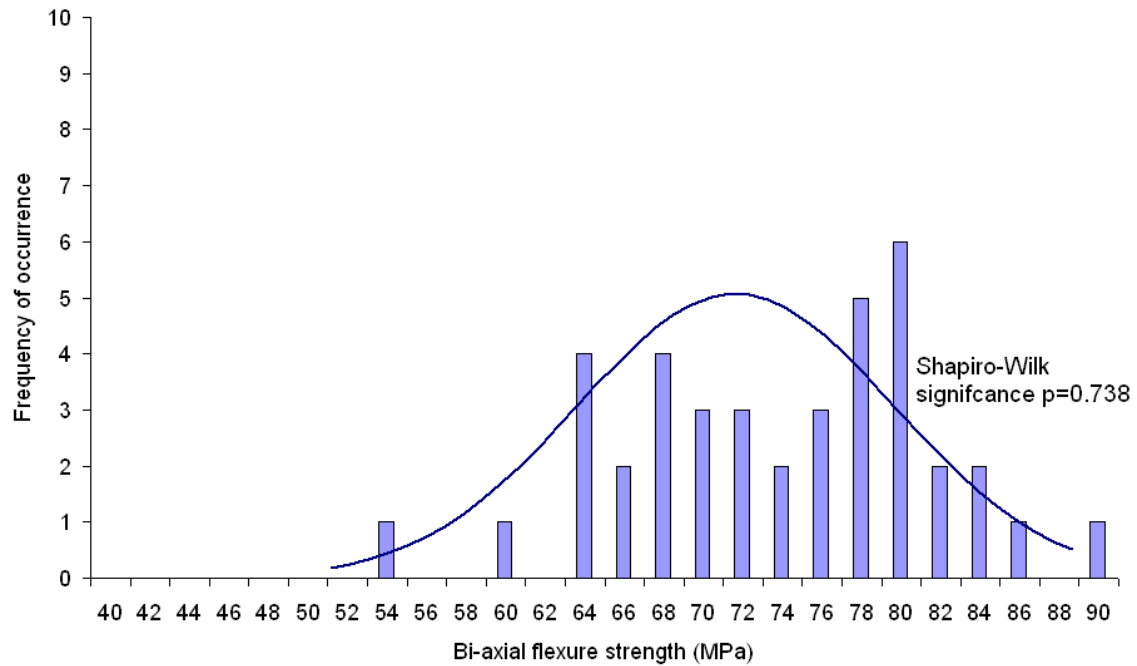


Figure 3a

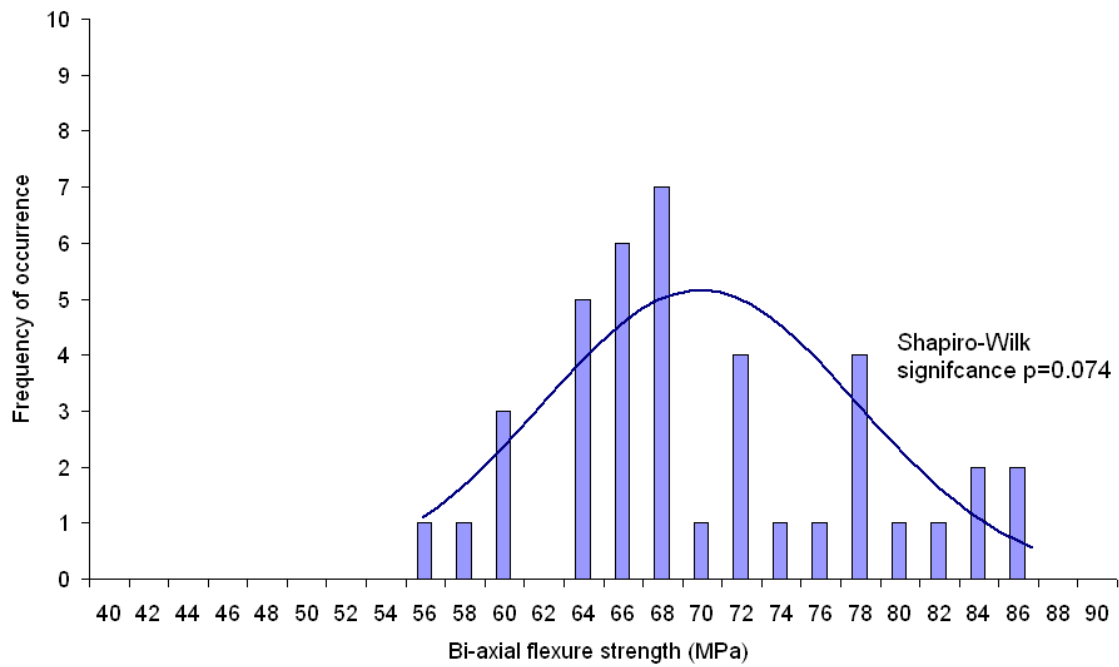


Figure 3b

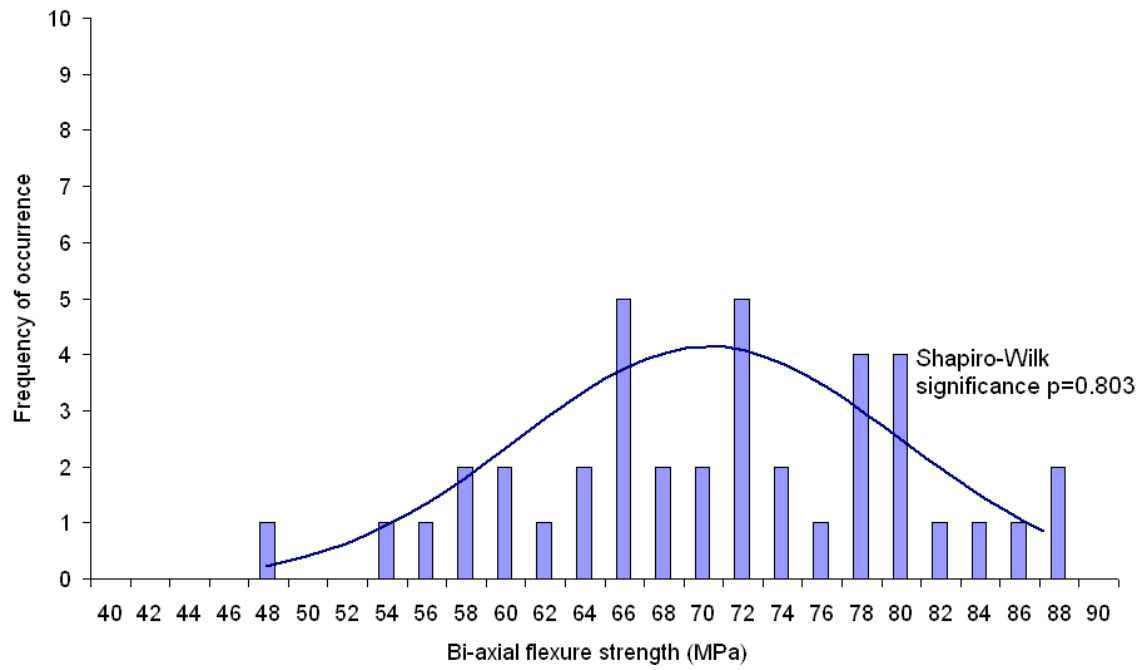


Figure 3c