©Operative Dentistry, 2006, **31-5,** 584-589

Effect of Early Water Exposure on the Strength of Glass Ionomer Restoratives

XY Wang • AUJ Yap • HC Ngo

Clinical Relevance

Modern day glass ionomer restoratives are not significantly weakened by early water exposure, after initial set.

SUMMARY

This study examined the effect of early water exposure on the shear strength of a spectrum of glass ionomer restoratives. The materials evaluated included conventional auto-cured (Fuji II [FT], GC), resin-modified light-cured (Fuji II LC [FL]) and, recently introduced, high strength auto-cured (Fuji IX GP Fast [FN], GC; Ketac Molar Quick [KQ], 3M-ESPE; Ketac Molar [KM], 3M-ESPE) cements. Sixteen specimens (8.7-mm in diameter and 1-mm thick) of each material were prepared in metal washers and randomly divided into 2 groups. The specimens were allowed to set for 6 minutes between polyester strips, to ensure completion of the initial set. The strips were subsequently removed, and the surfaces of Group 1 specimens were coated on both sides with resin (Fuji Coat LC, GC) and light cured for 10 seconds. Group 2 specimens were left uncoated. All specimens were then conditioned in distilled water at 37°C for 4 weeks. After conditioning, the specimens were restrained with a torque of 2.5 Nm and subjected to shear punch testing using a 2-mm diameter punch at a crosshead speed of 0.5-mm/minute. The mean shear strengths of the materials were computed and subjected to Independent Samples *t*-test and ANOVA/Scheffe's tests at significance level 0.05. Mean strength ranged from 78.34 to 99.36 MPa and 79.88 to 95.78 MPa for Groups 1 and 2, respectively. No significant difference in shear strength was observed between the 2 groups. For both groups, KM and KQ were significantly stronger than FT. Contrary to current teaching, early exposure to water did not weaken glass ionomer restoratives. A marginal increase in strength was actually observed for some materials.

*XY Wang, BDS, MD, lecturer, Department of Cariology, Endodontology and Operative Dentistry, Peking University School and Hospital of Somatology, PR China; research scholar, Department of Restorative Dentistry, Faculty of Dentistry, National University of Singapore, Republic of Singapore

Adrian UJ Yap, BDS, MSc, PhD, FAMS, FADM, FRSH, associate professor, Department of Restorative Dentistry, Faculty of Dentistry; assistant director, Centre for Biomedical Materials Applications and Technology, Faculty of Engineering, National University of Singapore, Singapore, Republic of Singapore

HC Ngo, BDS, MDS, associate professor, Colgate Australian Clinical Dental Research Centre, North Terrace Campus, The University of Adelaide, SA, Australia

*Reprint request: 22 Zhong Guan Cun South Avenue, Hai Dian District, Beijing, China 100081; e-mail: wangxiaoyanpx@gmail.com

DOI: 10.2341/05-106

INTRODUCTION

Glass ionomer cements were first introduced to the dental profession in the early 1970s.[1] They are derived from aqueous polymeric acids and a glass component, which is usually a fluoroalumino silicate. As filling materials, glass ionomer cements possess certain desirable properties, including chemical bonding to enamel and dentin, release of anticariogenic fluoride into adjacent

tooth structures and a low coefficient of thermal expansion similar to teeth. Glass ionomer cements are, however, susceptible to fracture and exhibit low wear resistance.[2] These deficiencies have limited their use to areas subject to low masticatory stresses (for example, Class V cavities) and as interim restorations in permanent teeth. In primary teeth, they are indicated for use in all cavity classes. The physico-mechanical properties of glass ionomer cements are dependent on the formation of a relatively insoluble polysalt matrix, which takes time to form. This matrix changes from being primarily soluble calcium polyacrylate based to a more stable aluminum polyacrylate over the first 24 hours.[3] If newly placed glass ionomer restorations are exposed to water too soon after placement, both water balance and setting reaction can be disturbed.[4] This results in erosion and water sorption,[5] which negatively affect translucency, strength and clinical performance.[6-8]

The application of different coatings (varnish, petroleum jelly, cocoa butter or light-cured resin) to the surface of glass ionomers after initial set has been recommended to overcome the problem of early moisture sensitivity.[3,7-8] The sealing of materials with coatings for at least 1 hour has been shown to produce specimens of optimum strength.[7] Among the various coating materials, light-cured unfilled resin gives the most efficient protection during cement maturation.[9-10] Resin-modified light-cured glass ionomers were subsequently introduced to help overcome the problems of "moisture sensitivity" and "low early mechanical strengths" associated with conventional auto-cured materials. Their formulation ranges from glass ionomer cements with the addition of a small quantity of resin components (for example, hydroxyethyl methacrylate [HEMA] or bisphenol A glycidyl methacrylate [BisGMA]) to more complex materials consisting of modified polyacid with light-polymerized side chains.[11] More recently, high strength auto-cured materials were developed and marketed. These materials have improved glass particle size and size distribution and increased glass surface reactivity. In addition, some or all of the calcium fluoroalumino silicate glass is replaced with strontium-based ones to increase radiopacity.[8] While these new materials have higher strength and more clinical applications, they do not have the translucency of regular restorative cements. Recent research on high strength auto-cured cements suggest early access to water will "positively influence" strength and hardness.[12-13] These studies, however, evaluated only 1 glass ionomer product (Fuji IX GP, GC Corporation, Tokyo, Japan). Further studies are required to determine whether the observations are applicable to other types of glass ionomer restoratives. As the application of resin coatings also incurs additional cost, clinical time and reduces fluoride release,[14-15] the effects of early water exposure on the strength of these cements warrants investigation.

The objective of this *in vitro* study was to examine the influence of early water exposure on the shear punch strength of conventional auto-cured, resin-modified light-cured and high strength auto-cured glass ionomer restoratives. The strength of materials with and without resin coating was also compared. It was hypothesized that early water exposure weakens glass ionomer cements.

METHODS AND MATERIALS

The glass ionomer restorative materials evaluated, their manufacturers, batch numbers and recommended setting time are shown in Table 1. The materials were supplied in capsulated form and activated/mixed according to the manufacturers' instructions. Shear punch specimens were made by injecting the cements into stainless steel washers (17.7-mm outer diameter, 8.7-mm inner diameter, 1-mm thick), which were supported by glass slides. A second glass slide was placed on top of the washers, and gentle pressure was applied to extrude excess material. Sixteen specimens were made for each material and randomly divided into 2 groups of 8. The resin-modified glass ionomer (Fuji II LC) was cured with a halogen curing light (Spectrum; Dentsply/Caulk, Milford, DE, USA) with a light exit window of 13-mm and a mean intensity greater than 400 mW/cm^2 for 20 seconds. All materials were allowed to set for 6 minutes between polyester strips to ensure completion of the initial set. Following removal of the strips, the surfaces of the Group 1 specimens were immediately coated on both sides with resin (Fuji Coat

Table 1: *The Different Glass Ionomer Restorative Materials Evaluated*

Materials	Classification	Manufacturer	Batch #3 (Shade)	Setting Time
Fuji II (FT)	Conventional, auto-cured	GC Corp, Tokyo, Japan	0304181 (21)	5 minutes, 30 seconds
Fuji II LC (FL)	Resin-modified, light-cure	GC Corp, Tokyo, Japan	0212135 (A2)	20 seconds (light cure)
Fuji IX GP Fast (FN)	High strength, auto-cured, fast set	GC Corp, Tokyo, Japan	0304032 (A2)	3 minutes
Ketac Molar Quick (KQ)	High strength, auto-cured, fast set	3M-ESPE, Seefeld, Germany	132967 (A2)	3 minutes, 30 seconds
Ketac Molar (KM)	High strength, auto-cured, regular set	3M-ESPE, Seefeld, Germany	114129 (A2)	4 minutes 30 seconds

LC, GC) and light cured for 10 seconds. Group 2 specimens were left uncoated. All specimens were then conditioned in distilled water at 37°C for 4 weeks.

Shear strength testing was conducted using a custom-designed micro-punch apparatus (Figure 1) mounted on an Instron Micro-tester (Model 5848, Instron Corp, Norwood, MA, USA). The thickness of each specimen was measured with a digital vernier caliper (Mitutoyo, Tokyo, Japan) with an accuracy of 0.01-mm prior to placement in the shear punch apparatus. The specimens were positioned in the apparatus via means of a self-locating recess, which provided a snug-fit, with the washers holding the specimens. The specimens were restrained by tightening a screw clamp to a torque of 2.5 Nm using a torque wrench. A tool steel punch with a flat end 2-mm in diameter was used to create shear force by sliding through a punch hole with a radial clearance of 0.01-mm. Prior to testing, the entire experimental setup, including the Instron machine and its 2kN load cell, was calibrated to ensure minimal frictional force as compared to the force value required to fracture the test specimens. Testing was done at a crosshead speed of 0.5-mm/minute and the maximum load was recorded. Shear strength was subsequently computed using the following formula:

$$\text{Shear strength (MPa)} = \frac{\text{Force (N)}}{\pi \times \text{Punch diameter (mm)} \times \text{Thickness of specimen (mm)}}$$

All statistical analysis was carried out at significance level 0.05. The effect of early water contact was established by comparing shear strength of resin-coated and uncoated specimens using Independent Samples *t*-test. One-way ANOVA and Scheffe's post-hoc tests were used to determine inter-material differences in strength. Differences in fracture modes between coated and uncoated specimens were analyzed using the Mann-Whitney test.

RESULTS

The mean shear strength of resin-coated (Group 1) and uncoated (Group 2) materials is shown in Table 2. Mean strength ranged from 78.34 to 99.36 MPa and 79.88 to 95.78 MPa for Groups 1 and 2, respectively. Independent Samples *t*-test revealed no significant difference in shear strength between the 2 groups. The results of inter-material comparison are reflected in Table 2. For Group 1, the shear strength of Ketac Molar and Ketac Molar Quick was significantly greater than Fuji II LC and Fuji IX GP Fast. Fuji II LC was significantly stronger than Fuji II. For Group 2, Ketac Molar, Ketac Molar Quick and Fuji II LC were significantly stronger than Fuji II. No significant difference in shear strength was observed between Fuji IX GP Fast and

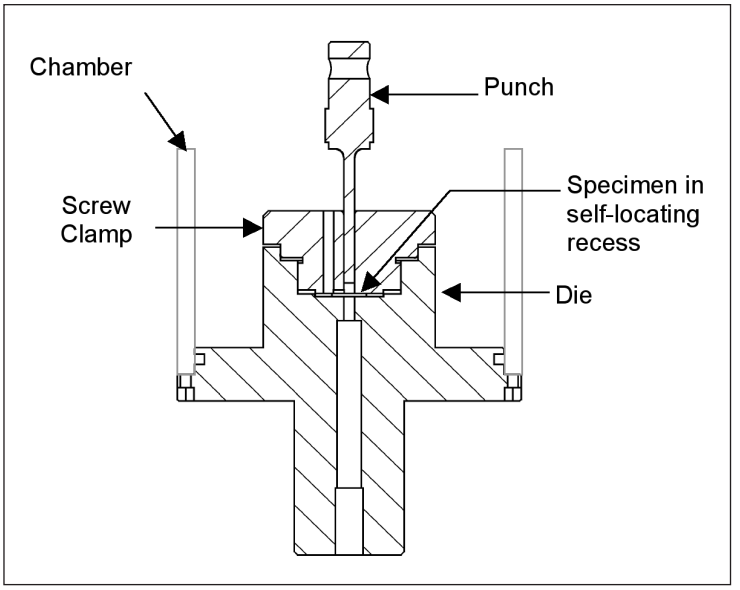

Figure 1: *Schematic representation of the micro-punch apparatus.*

Table 2: *Mean Shear Strength (MPa) of Resin-coated and Uncoated Materials*

Materials	Group 1 (Resin-coated)	Group 2 (Uncoated)
Fuji II (FT)	78.34 (7.52)[a]	79.88 (6.55)[a]
Fuji II LC (FL)	87.18 (3.78)[b]	92.75 (6.52)[b]
Fuji IX GP Fast (FN)	86.50 (6.48)[a,b]	88.40 (6.88)[a,b]
Ketac Molar Quick (KQ)	96.02 (3.75)[c]	94.71 (4.42)[b]
Ketac Molar (KM)	99.36 (4.10)[c]	95.78 (6.78)[b]

Standard deviations in parentheses

[a,b,c]: same letter indicates no significant difference. Results of one-way ANOVA/Scheffe's test (p<0.05).

Table 3: *Frequency Distribution of Failure Modes*

Materials	Group 1 (Resin-coated)		Group 2 (Uncoated)	
	Punch Out	Stress Fracture with Multiple Cracking	Punch Out	Stress Fracture with Multiple Cracking
Fuji II (FT)	6	2	7	1
Fuji II LC (FL)	8	0	8	0
Fuji IX GP Fast (FN)	2	6	3	5
Ketac Molar Quick (KQ)	7	1	6	2
Ketac Molar (KM)	6	2	1	7

Fuji II for both coated and uncoated groups. Fracture modes were divided into "punch out" and "stress fracture with multiple cracking," as described by Nomoto, Carrick and McCabe.[16] Frequency distribution of the 2 failure modes is shown in Table 3. With the exception of Ketac Molar, no significant difference in failure modes was observed between coated and uncoated specimens. For Ketac Molar, specimens that were resin-coated experienced significantly more circumferential cracking failures than those that were uncoated.

DISCUSSION

This study extended the work of Leirskar and others to more glass ionomer restoratives, including both auto and light-cured versions.[12] The International Standards Organization (ISO) has recommended different standard tests for auto-cured and resin-modified light-cured glass ionomers. Auto-cured materials are evaluated in compression, while light-cured materials are evaluated in fracture.[17-18] The difference in testing is due to technical difficulties faced when light curing compressive specimens and does not allow for comparison between glass ionomer cements. The shear punch test has been used for the standard testing of plastics and was recently advocated as a standard specification test across a broad range of restorative materials.[19,16] As occlusal or incisal forces during the masticatory cycle induce shear stresses in teeth and restorations, the shear punch test reflects qualities of clinical significance.[20] The advantages of the shear punch test have been reported by Nomoto and others.[16] The main distinction is the ease of preparing good quality specimens. For shear punch testing, the quality of the edges of the disc around the circumference has no direct influence on the testing outcome. While in flexural, compressive and diametral testing, the quality of the surfaces and edges of specimens is most critical. The only requirement for shear punch testing is that the 2 main faces of the disc specimens are flat and parallel.[16] The latter is greatly facilitated by the use of standard washers for specimen preparation. In this study, a torque of 2.5 Nm was applied to the specimens during shear punch testing as Nomoto, Carrick and McCabe reported significantly lower strength values were reported for specimens that were not restrained.[16] They hypothesized that unrestrained specimens are able to bend on application of the punch, creating localized stress concentration leading to premature failure. The reliability and reproducibility of the shear punch test was evidenced by the low standard deviations observed for all materials and treatment groups.

The strength of auto-cured cements has been shown to increase over a 1-year period.[21] A recent study found that the strength of a high strength auto-cured glass ionomer reached a maximum within 1 to 2 weeks for resin-coated and uncoated specimens, respectively.[12]

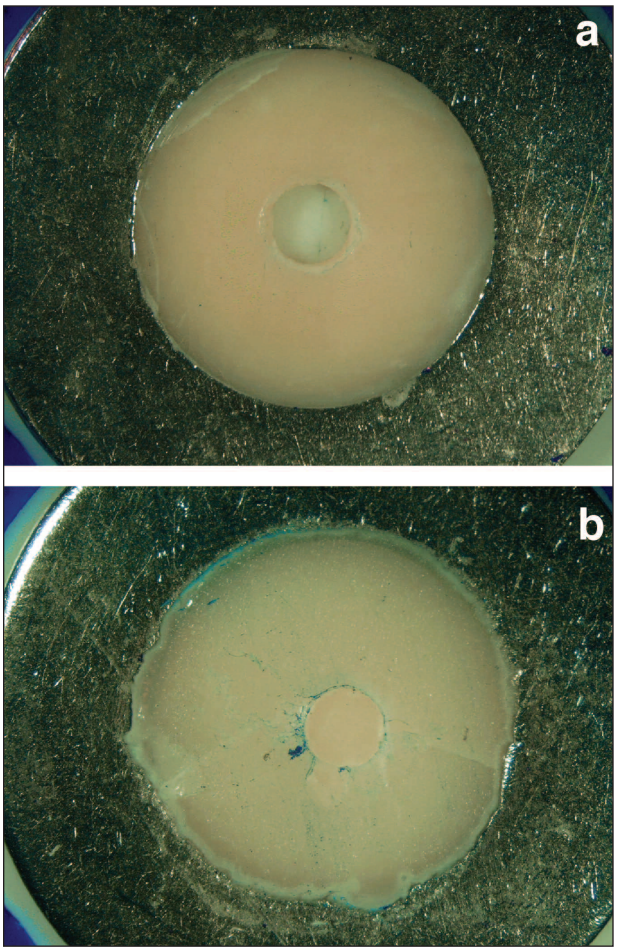

Figure 2: *Images of specimens showing (a) punch out and (b) stress fracture with multiple cracking failure modes.*

When resin-modified glass ionomers are immersed in distilled water, water uptake equilibrated within 2 to 3 weeks.[22] Because the strength of resin-modified cements peaked at 1 week and decreased after 1 month storage in an aqueous environment,[23] a 4-week conditioning period was selected. For all glass ionomer restoratives evaluated, no significant difference in strength was observed between resin-coated and uncoated specimens. These findings were contrary to the general instructions of manufacturers and previous studies on older generations of cements.[7-8] This may be attributed to advances in the method of manufacture and cement formulation. A previous study reported that the hardness of contemporary glass ionomers was not adversely affected by early exposure to water.[24] Leirskar and others found that early access to water increased the strength of a high strength auto-cured cement.[12] In this study, the authors found that early water contact did not positively or negatively influence the shear strength of glass ionomers. The apparent discrepancy may be attributed to the use of Fuji IX GP in Leirskar's study and Fuji IX GP Fast in the current study. Okada and others, who showed a significant increase in hard-

ness of high strength auto-cured cements following storage in both distilled water and human saliva, also employed the use of Fuji IX GP.[13] The strength of glass ionomer cements has been associated with an increase in "bound" water.[25-26] As the hydration of glass ionomers increase with age, so does strength; Leirskar and others suggested that resin coating limits the hydration process interfering with the steady increase in strength. Fuji IX GP Fast is an improved version of Fuji IX GP.[12] The setting time of Fuji IX Fast is half that of Fuji IX GP. This was attributed to the use of smaller glass particles (mean particle size: Fuji IX GP–13.43 µm; Fuji IX GP Fast–7.13 µm) and a higher powder:liquid weight ratio (Fuji IX GP–0.35:0.[10]; Fuji IX GP Fast–0.36:0.10) in Fuji IX Fast.[27] The shortened maturation time of Fuji IX GP Fast may make it less susceptible to the effect of early water exposure. Although the strength of the glass ionomers evaluated was not influenced by early water contact, translucency may be decreased and warrants further investigation. While this may not be important for high strength auto-cured cements, which are opaque in nature and used for the restoration of posterior teeth, it is important for resin-modified materials that are frequently employed to restore front teeth.

For both the resin-coated and uncoated groups, Ketac Molar, Ketac Molar Quick and Fuji II LC were significantly stronger than Fuji II. The higher strength of the resin-modified (Fuji II LC), when compared to its conventional auto-cured counterpart (Fuji II), was in agreement with previous studies.[11] The mechanical properties of high strength auto-cured materials were product- and treatment-specific. No significant difference in strength was observed between Fuji IX GP Fast and Fuji II for both groups. This may be explained in part by the use of smaller, irregular shaped particles in Fuji IX Fast, which could increase the risk for local stress concentrations leading to local crack growth and decreased strength.[28] Although Ketac Molar and Ketac Molar Quick were significantly stronger than Fuji IX Fast when specimens were resin-coated, no significant difference in strength was observed between these materials when specimens were uncoated and exposed to water after initial set. The relative performance of Fuji IX Fast can therefore be enhanced by not protecting the cement with a resin coating. With the exception of Fuji IX Fast and Ketac Molar, all materials failed predominantly in a "punch out" or shear mode, and the failure mode was irrespective of early water contact. For Fuji IX Fast, tensile failure (stress fracture with multiple cracking) was observed for the majority of specimens in both treatment groups. Ketac Molar specimens failed predominantly in shear when coated, and in tensile, when uncoated. The clinical significance of this observation is not known and should be explored.

CONCLUSIONS

Early access to water did not negatively influence the strength of glass ionomer restoratives. Contrary to the instructions issued by most manufacturers, there is no need for placement of a resin coating over high strength auto-cured cements, unless they require protection from dehydration. Although the strength of resin-modified light-cured and modern conventional auto-cured materials is not affected by early water contact, resin coating is still advocated until the effects of early water contact on aesthetic properties is determined.

Acknowledgements

The authors thank Ms SH Lee and Ms QH San for their assistance in conducting the experiment. This paper was supported by NUS ARF Grant R-224-000-014-112.

(Received 11 July 2005)

References

1. Wilson AD & Kent BE (1972) A new translucent cement for dentistry: The glass ionomer cement *British Dental Journal* **132(4)** 133-135.
2. McLean JW (1988) Glass ionomer cements *British Dental Journal* **164(9)** 293-300.
3. McLean JW & Wilson AD (1977) The clinical development of the glass-ionomer cements. 1. Formulations and properties *Australian Dental Journal* **22(1)** 31-36.
4. Mount GJ (1997) Longevity in glass-ionomer restorations: Review of a successful technique *Quintessence International* **28(10)** 643-650.
5. Mount GJ & Makinson OF (1982) Glass-ionomer restorative cements: Clinical implications of the setting reactions *Operative Dentistry* **7(4)** 134-141.
6. Asmussen E (1983) Opacity of glass ionomer cements *Acta Odontologica Scandinavica* **41(3)** 155-157.
7. Causton BE (1981) The physico-mechanical consequences of exposing glass ionomer cements to water during setting *Biomaterials* **2(2)** 112-115.
8. Mount GJ (1999) Glass ionomers: A review of their current status *Operative Dentistry* **24(2)** 115-124.
9. Hotta M, Hirukawa H & Yamamoto K (1992) Effect of coating materials on restorative glass-ionomer cement surface *Operative Dentistry* **17(2)** 57-61.
10. Ribeiro AP, Serra MC, Paulillo LA & Rodrigues AL Jr (1999) Effectiveness of surface protection for resin-modified glass-ionomer materials *Quintessence International* **30(6)** 427-431.
11. Sidhu SK & Watson TF (1995) Resin-modified glass ionomer materials. A status report for the American Journal of Dentistry *American Journal of Dentistry* **8(1)** 59-67.
12. Leirskar J, Nordbø H, Mount GJ & Ngo H (2003) The influence of resin coating on the shear punch strength of a high strength auto-cure glass ionomer *Dental Materials* **19(2)** 87-91.

13. Okada K, Tosaki S, Hirota K & Hume WR (2001) Surface hardness change of restorative filling materials stored in saliva *Dental Materials* **17(1)** 34-39.
14. Hattab FN & Amin WM (2001) Fluoride release from glass ionomer restorative materials and the effects of surface coating *Biomaterials* **22(12)** 1449-1458.
15. Mazzaoui SA, Burrow MF & Tyas MJ (2000) Fluoride release from glass ionomer cements and resin composite coated with a dentin adhesive *Dental Materials* **16(3)** 166-171.
16. Nomoto R, Carrick TE & McCabe JF (2001) Suitability of a shear punch test for dental restorative materials *Dental Materials* **17(5)** 415-421.
17. *International Organization for Standardization* (2003) Specification for dental materials—water-based cements ISO 9917.
18. *International Organization for Standardization* (1999) Specification for light-activated water-based cements ISO 9917-2.
19. *American Society for Testing and Materials Standards* (1993) Standard test method for shear strength of plastics by punch tool ASTM D732-6.
20. Roydhouse RH (1970) Punch-shear test for dental purposes *Journal of Dental Research* **49(1)** 131-136.
21. Crisp S, Lewis BG & Wilson AD (1976) Characterization of glass-ionomer cements. 1. Long term hardness and compressive strength *Journal of Dentistry* **4(4)** 162-166.
22. Kanchanavasita W, Anstice HM & Pearson GJ (1997) Water sorption characteristics of resin-modified glass-ionomer cements *Biomaterials* **18(4)** 343-349.
23. Yap AU, Mudambi S, Chew CL & Neo JC (2001) Mechanical properties of an improved visible light-cured resin-modified glass ionomer cement *Operative Dentistry* **26(3)** 295-301.
24. Khouw-Liu VH, Anstice HM & Pearson GJ (1999) An *in vitro* investigation of a poly (vinyl phosphonic acid) based cement with four conventional glass-ionomer cements. Part 2: Maturation in relation to surface hardness *Journal of Dentistry* **27(5)** 359-365.
25. Wilson AD, Paddon JM & Crisp S (1979) The hydration of dental cement *Journal of Dental Research* **58(3)** 1065-1071.
26. Wilson AD, Crisp S & Paddon JM (1981) The hydration of a glass ionomer (APSA) cement *British Polymer Journal* **13(1)** 66-70.
27. Yap AU, Pek YS & Cheang P (2003) Physico-mechanical properties of a fast-set highly viscous GIC restorative *Journal of Oral Rehabilitation* **30(1)** 1-8.
28. Yap AU, Lee MK, Chung SM, Tsai KT & Lim CT (2003) Effect of food-simulating liquids on the shear punch strength of composite and polyacid-modified composite restoratives *Operative Dentistry* **28(5)** 529-534.