A COMPARATIVE STUDY OF PHYSICAL PROPERTIES OF GIC CONTAINING β-MONOCALCIUM SILICATE TO MINERAL TRIOXIDE AGGREGATE (MTA)

A THESIS

BY

SIRIJIT POTHIRAKSANONT

Presented in Partial Fulfillment of the Requirements for the

Master of Science Degree in Endodontology

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Mineral trioxide aggregate(MTA) is currently a material of choice for various endodontic treatments, but its lacking reasonable setting time and preferable handling difficulty prompted researchers to look for an alternative. This study investigated the setting time, pH and compressive strength of a novel cement ; GIC containing monocalcium silicate(GIC-CS) in three different ratios (10%, 30% and 50%CS by weight) by comparing with White ProRoot® MTA and GIC (Ketac™Molar). Methods: Six specimens in each group were used to measure initial and final setting times according to ISO 6876 methods. Measurement of pH were collected from supernatant of each crushed material from 10 minutes to 48 hours period, this test was done in triplicate. Six specimens in each groups were prepared for compressive strength evaluation in each time period; following the ISO 9917 method. The strength was measured at 1,3,7,21,28 days. The results showed that both initial and final setting times of all GIC-CS groups (initial: 4.42±0.2-8.75±0.67, final: 5.50±0.35 - 12.88±0.59 min.) were significantly less than that of MTA (initial:63.67±2.42, final: 121.5±2.66min) ($p < 0.05$). Adding of CS into GIC significantly neutralize the pH of GIC in the 48 hours period ($p < 0.05$). Compressive strength of CS10 and CS30 groups showed no statistically significant different from GIC group. CS50 group showed statistically reduction in compressive strength compared with GIC ($p < 0.05$) at 21 and 28 days periods. However; this reduction was not shown to be clinically relevant. In conclusion, by adding of 10%, 30% and 50% of CS into GIC exhibited significantly shortened setting time when compared with MTA, neutralized the pH of GIC and not clinically affected to GIC's compressive strength. This material could potentially developed further to use in endodontics.

การศึกษาเปรียบเทียบคุณสมบัติทางกายภาพของวัสดุกลาสไอโอโนเมอร์ซีเมนต์ที่มีส่วนประกอบของเบต้าโมโนแคลเซียมซิลิเกตกับวัสดุมิเนอร์รัลไตรออกไซด์แอกกรีเกต (MTA)

บทคัดย่อ

ของ

ศิริจิต โพธิรักษานนท์

เสนอต่อบัณฑิตวิทยาลัย มหาวิทยาลัยศรีนครินทรวิโรฒ เพื่อเป็นส่วนหนึ่งของการศึกษา

ตามหลักสูตรวิทยาศาสตรมหาบัณฑิต สาขาวิชาวิทยาเอนโดดอนต์

กันยายน 2556

ศิริจิต โพธิรักษานนท์ . (2556). *การศึกษาเปรียบเทียบคุณสมบัติทางกายภาพของวัสดุกลาสไอโอโนเมอร์ซีเมนต์ที่มีส่วนผสมของเบต้าโมโนแคลเซียมซิลิเกตกับวัสดุมิเนอร์รัลไตร ออกไซด์แอกกรีเกต (MTA).* ปริญญานิพนธ์ วท .ม.(วิทยาเอนโดดอนต์). กรุงเทพฯ : บัณฑิตวิทยาลัย มหาวิทยาลัยศรี นครินทรวิโรฒ . คณะกรรมการควบคุม : ทพญ.ดร.จารุมา ศักดิ์ดี , ผศ.ดร. ปุณณมา ศิริพันธ์โนน.

มิเนอร์รัลไตรออกไซด์แอกกรีเกต (MTA) จัดเป็นวัสดุที่ได้รับความนิยมในการใช้งานทางเอนโดดอนติกส์อย่างแพร่หลาย อย่างไรก็ตาม MTA มีข้อเสียที่สำคัญคือคือ มีระยะเวลาในการก่อตัวยาวนานและ ลักษณะ การใช้งานที่ยากลำบากจึงทำให้นักวิจัยพยายามที่จะพัฒนาวัสดุมาทดแทน การศึกษานี้มีวัตถุประสงค์ เพื่อเปรียบเทียบคุ ณสมบัติทางกายภาพอันได้แก่ ระยะเวลาในการก่อตัว , ค่าความเป็นกรด-เบส และ ความทนแรงอัดของวัสดุกลาสไอโอโนเมอร์ซีเมนต์ที่มีส่วนผสมของเบต้าโมโนแคลเซียมซิลิเกต (GIC-CS) ที่อัตราส่วนต่างๆ 3 อัตราส่วนคือ ร้อยละ 10 (CS10), ร้อยละ30 (CS30), และร้อยละ 50 (CS50) โดยน้ำห นัก กับวัสดุ กลาสไอโอโนเมอร์ซีเมนต์ ดั้งเดิม (Ketac™ Molar) และ ProRoot® MTAชนิดสีขาว โดยเตรียมชิ้นงานกลุ่มละ 6 ชิ้นในการวัดระยะเวลาก่อตัวของซีเมนต์โดย วัดระยะเวลาการก่อตัวโดยเข็มแบบกิลมอร์ประยุกต์ตามมาตรฐาน ISO 6876 วัดค่าความเป็นกรด-เบส โดยใช้เครื่องวัดความเป็นกรด-เบสวัดที่เวลาหลังผสมสาร 10นาทีจนถึง 48 ชั่วโมงโดยทำการทดลองกลุ่มละ 3 ครั้ง และเตรียมชิ้นงานกลุ่มละ 6 ชิ้นวัดค่าความทนแรงอัดในแต่ละช่วงเวลาที่ระยะเวลา 1,3, 7,21 และ 28วันโดยประยุกต์ตามมาตรฐาน ISO 9917 ผลการศึกษาพบว่าระยะเวลาการก่อตัวของซีเมนต์ GIC-CS (initial: 4.42±0.2-8.75±0.67, final: 5.50±0.35 - 12.88±0.59 min.) มีค่าน้อยกว่าระยะเวลาก่อตัวของ MTA (initial:63.67±2.42, final: 121.5±2.66min) อย่างมีนัยสำคัญทางสถิติ ($p<0.05$) เมื่อเติมโมโนแคลเซียมซิลิเก ต (CS)ลงไปใน GIC ส่งผลให้ค่าความเป็นกรด- เบสเป็นกลางมากขึ้นอย่างมีนัยสำคัญทางสถิติ ($p<0.05$) ค่าความทนแรงอัดของวัสดุ CS10, CS30 นั้นไม่แตกต่างจากกลุ่ม GIC อย่างมีนัยสำคัญทางสถิติ ($p>0.05$)ในทุกช่วงเวลา ถึงแม้กลุ่ม CS50 จะพบค่าความทนแรงอัดลดลงจากกลุ่ม GIC อย่างมีนัยสำคัญทางสถิติ ($p<0.05$)ที่ระยะเวลา 21และ28 วัน แต่ไม่ส่งผลกระทบต่อการนำมาใช้งานทางคลินิก สรุปได้ว่าวัสดุกลาสไอโอโนเมอร์ซีเมนต์ที่มีส่วนผสมของเบต้าโมโนแคลเซียมซิลิเกต (GIC-CS) มีระยะเวลาการก่อตัวน้อยกว่า MTA อย่างมีนัยสำคัญ และยังช่วยให้ความเป็นกรดของซี เมนต์หลังผสมลดลง นอกจากนั้นก็ไม่มีผลกระทบทางคลินิกต่อความทนแรงอัดของวัสดุกลาสไอโอโนเมอร์ที่ระยะเวลา 28วัน วัสดุ GIC-CS จึงอาจจะนำมาพัฒนาใช้ในงานทางเอนโดดอนติกส์ได้ในอนาคต

The thesis titled

" A Comparative Study of Physical Properties of GIC containing β-monocalcium silicate to Mineral Trioxide Aggregate(MTA)"

By

Sirijit Pothiraksanont

Has been approved by the Graduate School as partial fulfillment of the requirements for the Master of Science degree in Endodontology of Srinakharinwirot University

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Sirijit Pothiraksanont.

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LIST OF ABBREVIATIONS

MTA	Mineral trioxide aggregate
WMTA	White mineral trioxide aggregate
GMTA	Grey mineral trioxide aggregate
PC	Portland cement
C-S-H	Calcium silicate hydrate gel
GIC	Glass ionomer cement
CS	Monocalcium silicate
W	Wollastonite
ps-W	PseudoWollastonite
HA	Hydroxyapatite

CHAPTER 1

INTRODUCTION

Mineral trioxide aggregate (MTA) cement, categorized as calcium silicate-based cements, have been proven to be bioactive and biocompatible.[1] MTA is successfully utilized for various endodontic therapies such as pulp capping, perforation repair, root-end filling, forming an apical barrier , and a root canal filling.[1-3] However, MTA still has some disadvantages including its handling difficulty, prolonged setting time [4, 5] and relatively high cost.

MTA consists primarily of tricalcium silicate (C_3S/Ca_3SiO_5) and dicalcium silicate (C_2S/Ca_2SiO_4) which are the major constituents responsible for the cement's strength and bioactivity after hydration.[6] Several studies have investigated the properties of both calcium silicates modified by many methods, in hopes of overcoming MTA's drawbacks.[7, 8] However, none of the developed materials so far has yet fulfilled the ideal properties.

Monocalcium silicate($CS/CaSiO_3$)/ Wollastonite(W) ceramic is one of the promising bioactive bone cements. It has been widely recommended for decades as a bone substitute. [9] Ni *et al.* [10]studied the behaviour of osteoblast cells on W glass surfaces and found that differentiation and proliferation of osteoblasts were improved when exposed to these materials. Monocalcium silicate has an ability to form a hydroxyapatite (HA) layer on its surface when comes into contact with simulated body fluid, faster than any other form of calcium silicate.[11-14] This material is considered novice in dentistry. Shie *et al*.[15] in 2012 found that higher ratio of silicate/calcium content in calcium silicate cement improved the attachment of cells and their proliferation. In this study, monocalcium silicate was added to glass ionomer cement in order to benefit from its self-setting through an acid-base reaction.

Glass ionomer cements(GICs) have been used as luting and filling materials in dentistry for more than 40 years.[16] GICs are set by an acid-base reaction between fluoroaluminosilicate glass and polyalkenoic acids. The outstanding advantages of glass ionomer cements over other materials are their ability to chemically bond to tooth structure, and its cariostatic effect from released fluoride.[17] However, these materials are limited in their applications due to low compressive strength, brittleness, and low wear resistance.[18] They also project more cytotoxicity to PDL cell than MTA in cell culture technique.[19] There are many types of GICs available in the market; such as conventional GICs, resin-modified GICs, hybrid GICs, Tri-cure GICs and metal reinforced GICs/cermets, each of which has been developed in order to suit specific objective. Among all types of GICs ,Costa *et al*.[20] found that conventional glass-ionomer cement especially Ketac™ Molar projected the least cytotoxicity to odontoblast cell line.[19]

Modification of GICs by adding bioactive particles to improve biocompatibility and physical strength has been in the field of interest.[17, 21-23] For this reason, the new material; GIC containing monocalcium silicate (GIC-CS), was developed aiming to combine the good characteristics of both materials together.

Therefore, the objective of this study was to investigate the physical and mechanical properties of GIC-CS compound comparing with conventional Ketac™ Molar and ProRoot® MTA by means of setting time, pH and compressive strength.

Research question:

Do GIC containing monocalcium silicate (GIC-CS) compound has the same physical and mechanical properties as ProRoot MTA and Ketac™ Molar?

Research objectives:

To examine the physical and mechanical properties of GIC containing monocalcium silicate (GIC-CS) compound and compare them to ProRoot MTA and Ketac™ Molar in terms of setting time, pH value and compressive strength

Hypothesis:

Null hypothesis(H_0):

The physical properties of the GIC-CS compound are not different from those of ProRoot MTA.

The physical properties of the GIC-CS compound are not different from those of and Ketac™ Molar.

Alternative hypothesis(H_1)

-The physical properties of the GIC-CS compound are different from those of ProRoot MTA.

-The physical properties of the GIC-CS compound are different from those of Ketac™ Molar.

Keywords:

Wollastonite, biomaterial, Ketac molar , MTA, monocalcium silicate, physical property, compressive strength, setting time, pH

Research design:

Laboratory experimental research

Limitations:

This research is an *in vitro* study which may not represent clinical situation

Benefits:

If the GIC-CS compound shows comparable physical and mechanical properties to ProRoot® MTA, it could be an alternative material to ProRoot MTA for various dental applications.

CHAPTER 2
LITERATURE REVIEW

Vital pulp therapy and regenerative endodontics have received much attention as a new trend in maintaining pulp vitality and function. One of many important factors that determine success of vital pulp therapy is the type of material used. The ideal material for pulp capping should be biocompatible to pulp tissue. It should also promote the formation of dentin, provide bacterial tight seal, and have high compressive strength. [24]

Mineral Trioxide Aggregate (MTA) has been successfully used in vital pulp therapy and regenerative endodontics. Although, MTA has become very popular due to its excellent biocompatibility and sealing ability[25], it has a few flaws which are difficult handling characteristic, extended setting time and relatively expensive. An innovation to provide ideal capping agent is not yet to be found. Among all of the GICs, Ketac™ Molar was found to be the least cytotoxic material,[20] and possessed highest compressive and flexural strength.[26, 27] This literature review includes MTA; the existing pulp capping material, and the material in question, Ketac™ Molar and monocalcium silicate/Wollastonite.

1.MTA

In 1993, Mineral trioxide aggregate (MTA) was first used in dentistry as root-end filling and repair material.[28, 29] In 1995, it was patented in the United States as Patent Numbers 5,769,638 and 5,415,547. Subsequently in 1998, MTA was approved for endodontic applications as ProRoot ® MTA (Dentsply Tulsa Dental, Tulsa, OK, USA) in grey colored formula (GMTA). MTA has been investigated later as pulp-capping agents, pulpotomy dressing materials, and material for treatment of immature apices and root canal sealers.[28, 30, 31]

In 2002, the tooth colored formula or white MTA (WMTA) had substituted the GMTA for esthetic reason. Both types of MTA had similar chemical constitutions except the white MTA did not contain tetracalcium aluminoferrite, which causes intensive staining.[32] Results from both X-ray energy dispersive analysis and X-ray diffraction analysis confirmed upon this dissimilarity between two types of MTA.(33) The comparison of elemental compositions by electron probe microanalysis showed that GMTA had higher concentrations of Al_2O_3, MgO and significantly FeO(which was 10 times higher) (34) compared to that of WMTA. Investigation in particle sizes by Asgary et al.[34] also demonstrated a smaller particle size of WMTA than that of GMTA. This means that WMTA could provide more surface area for hydration reactions and give greater early strength.(6) In addition, a finer particle size resulted in a smoother surface and caused less irritation to living tissues.(35) A study by Oviir *et al.*(36) showed that OCCM-30 cementoblast and OKF6/TERT1 keratinocytes preferred to grow on the surface of WMTA to that of GMTA. When exposed to WMTA, cell proliferation increased significantly.(36, 37)

MTA and Portland cement (PC) showed many similarities in chemical and physical characteristics. (33, 34) ProRoot® MTA is essentially 75 wt % PC, 20 wt% bismuth oxide, and 5 wt% calcium sulfate. Calcium sulfate acts as a setting modifier. Bismuth oxide is added for radiopacity to allow for a radiographic assessment.(33, 38) The primary component, Portland cement, has 4 main components. (Table1)

TABLE 1 MAIN COMPONENTS OF PORTLAND CEMENT(PC).(6, 39)

Components	Chemical formula	Abbreviation	%wt
Tricalcium silicate	$3CaO \cdot SiO_2$ / Ca_3SiO_5	C_3S	55%
Dicalcium silicates	$2CaO \cdot SiO_2$ / Ca_2SiO_4	C_2S	20%
Tricalcium aluminate	$(CaO)_3 \cdot Al_2O_3$	C_3A	10%
Tetracalcium aluminoferrite	$(CaO)_4 \cdot Al_2O_3 \cdot Fe_2O_3$	C_4AF	10%

Tricalcium silicate is the most reactive constituent of Portland cement. It is the first component to react with water and form calcium silicate hydrate (C-S-H), giving early strength to Portland cement. However, a study has shown that dicalcium silicate has better long-term strength contribution.(6)

Another remarkable advantage of MTA over other materials available is its superior ability to induce the formation of bone-like apatite by allowing bone cells proliferation , differentiation and attachment of bone cells on its surface.[25, 40, 41] These osteoinductive and osteoconductive characteristics have been attributed to hydroxyapatite formation that triggered by silanol (Si-OH) group on MTA's surface and accelerated by the continuous release of calcium ions from MTA due to the interaction of MTA with phosphate in the tissue fluid.[40, 42]

Moreover, MTA has been proven to be biocompatible to several types of cells including bone and PDL cells in previous studies. Koh *et al*.[41] in 1998 studied the effect of MTA to MG-63 osteosarcoma cells, and found good attachment to the material. Mitchell *et al*. in 1999[43] found that MG-63 osteosarcoma cells attached over three variant formulations of MTA and suggested to safely use in clinical situations.[43] Conformed with Perinpanayagam and Al-Rabeah[44] in 2009 who studied the effect of MTA on human primary alveolar bone cells, and found Runx2 (Runt-related transcription factor 2: predominant osteoblast differentiation gene) expression in osteoblasts. Several studies have shown that MTA was significantly less toxic than other root-end filling materials when freshly mixed, and then cytotoxicity was reduced when fully set at 24 hours.[41, 43]

1.1 The physical and mechanical properties of MTA

The physical and mechanical properties of MTA vary due to many variables such as the particle size, powder to liquid ratio, temperature, water presence, mixing method, the pH value of the environment and the type of MTA.[6, 32, 45] Fridland and Rosado [46]believed that some of these factors cannot be controlled easily; therefore, different results might be obtained during a study on physical properties of MTA. As manufacturer recommended, MTA is mixed with supplied sterile water in a 3:1powder to liquid ratio.[28] The material is kept in contact with a moist cotton pellet until the next use, to avoid its hydrating setting reaction.[6] MTA is also known as "Hydraulic Silicate Cement" by Darvell and Wu in 2011.[47] In other words, moisture is required to set MTA as MTA powder contains tiny hydrophilic particles that form calcium silicate hydrate gel(C-S-H) and calcium hydroxide($CaOH_2$) or portlandite [48] in the presence of moist and become solid.[33] C-S-H is the principal skeletal of hydration product of calcium silicate.[6] Tricalcium silicate reacts to form C-S-H almost immediately during the first wetting but this C-S-H has very poor crystallinity, and only a few broad, weak bands can be detected by the X-ray diffraction (XRD) patterns.[6]

The initial setting time of White MTA (WMTA) was reported to be 45 minutes, while the final setting times was found to be 140 minutes.[49] The average setting time of WMTA was 2 hours 45 min (± 5 min), which is longer than that of amalgam, IRM, and Super-EBA.[50] For GMTA, its initial and final setting times are significantly greater than that of WMTA.[4, 49] Portland cement (PC) takes at least 4 hours to achieve a final set. MTA and PC contain dehydrated calcium sulfate, added by the manufacturer to control the rate of reaction.[49] In a study by Lee *et al.*[51] in 2004, a lower environmental pH (pH 5) retarded the dissolution of certain components in MTA, such as C_3S, C_2S and C_3A, and thus inhibited the hydration reaction of MTA. To decrease MTA setting time and improve its handling characteristic, various hydration accelerators were added to the mixture of mineral trioxide aggregate.[7, 52, 53] An investigation on the effect of $CaCl_2$ on the setting time of Ca_3SiO_5 by Wang *et al.* [7] in 2008 demonstrated a decrease in both the initial (I) and final (F) setting times from 90 to 50 minutes, and 180 to 90 minutes respectively. In a study by Kogan *et al.*,[52] the setting time of GMTA was reduced to 20- 25 minutes if NaOCl gel, K-Y jelly (Johnson & Johnson, Inc., New Brunswick, NJ, USA), and 5% $CaCl_2$ were used as additives. However, the compressive strength was also found to be much lower than when MTA was mixed with water. In 2011, AlAnezi *et al.*[53] also reported that the addition of KY liquid, $CaCl_2$, and NaOCl to GMTA decreased its setting time and improved its handling characteristics. Moreover, osteoblasts and fibroblasts cell line remained the same biocompatibility to GMTA even when it was mixed with additives (Table2). Lee *et al.*[54] in 2011 found that calcium lactate gluconate (CLG); another hydration accelerators, also improved the setting time but impaired the compressive strength of MTA.

TABLE 2 SETTING TIMES AND COMPRESSIVE STRENGTH FOR GREY MTA MIXED WITH VARIOUS ADDITIVES.[52, 53]

Additive	Kogan *et al.*2006		AlAnezi *et al.*2011
	Setting time** (min)	compressive strength (MPa± SD)	Setting time# (mean ± SD)
Sterile water (control)	50	28.4 ± 8.2	195 ± 18.027
Chlorhexidine gluconate gel N/A (Consepsis V; Ultradent Products, Inc., South Jordan, UT, USA)	did not set until the end of the observation period (4 h)	N/A	-
NaOCl gel (ChlorCid V; 20 Ultradent Products, Inc.)	20*	17.1±3.8	130±13.22
K-Y jelly (K-Y; Johnson & Johnson, 20 Inc., New Brunswick, NJ, USA)	20*	18.3±3.4	70±13.22*
2% lidocaine HCl with 1:100,000 epinephrine	120	32.6±12.7	-
Saline	90	39.2±7.5	-
3% $CaCl_2$	50	19.3±3.7	-
5% $CaCl_2$	25*	19.6±2.9	123.33±2.88*

* $P < .05$, Adapted from Kogan *et al.*[52] (**Vicat apparatus) and AlAnezi *et al.*[53] (#Gilmore apparatus)

In leakage tests, MTA has been shown to have less leakage, and it can adapt far better to the dentinal walls at the root apex than amalgam, super EBA or IRM.[28, 55, 56] The better sealing ability of MTA may be because it expands slightly upon setting. There are still controversy regarding the setting expansion between GMTA and WMTA. Matt *et al.*[57]found that the original version of WMTA demonstrated significantly more leakage than GMTA in immediate apical barriers. The manufacturer reformulated the product with reduced particle size in 2003 to improve its biomechanical properties. The improved WMTA showed similar leakage properties to GMTA when tested in leakage studies.[4, 49, 58, 59] However, Al-Hezaimi *et al.* [60]used a saliva leakage model, and found more saliva leakage when sealed with WMTA compared with GMTA. Storm *et al.*[61] compared the hydroscopic linear setting expansions of GMTA, WMTA, and PC. They also found a significantly greater expansion for GMTA than for either WMTA or Portland cement. Linear setting expansion values for GMTA, WMTA and PC were 1.02%, 0.08% and 0.29% respectively.[61]

A study by Hawley *et al*.[62] found no significant difference in the setting expansions of both GMTA and WMTA when the water to powder ratios were varied. GMTA; however, expanded significantly greater than WMTA.

The alkalinity of MTA after mixing was well known. Right after mixing, the pH of MTA was around 10.2. It then rose to 12.5 in approximately 3 hours and remained at that level thereafter.[50] Its alkalinity was due to the production of calcium hydroxide[$Ca(OH)_2$] as a by-product after hydration of calcium silicate-based cement.[33] This has an important role for the cement being highly alkaline pH[50] which leads to its antibacterial, anti-inflammatory properties[7] and biocompatibility. Materials of the same composition with an addition of a latent hydraulic binder, that reacts with calcium hydroxide, were shown to have reduced cell growth and proliferation.[63] Calcium hydroxide production also plays an important role in making MTA become bioactive material due to its apatite-forming ability.[48]

The radiopacity of MTA was equivalent to 6.4 mm[32] -7.17 mm[50] of aluminum step wedge. For dentin, gutta percha, and amalgam, their radiopacity were 0.7 mm Al, 11.0 mm Al, and 15. 6 mm Al respectively.[64]

As a pulp capping material, MTA should provide such high compressive strength in order to withstand occlusal force. MTA's mean compressive strength is ranged from 35-50MPa at 24 hours after mixing.[4, 50] Its mean compressive strength is significantly less than other root-end filling materials'; such as amalgam, IRM, and Super EBA, at 24 hours (40±5 MPa) then rise to the same strength after 21 days at 67.3±6.6 MPa).[50] The compressive strength of MTA reaches the maximum values several days after mixing is owing to its setting reaction of the main constituents; tricalcium and dicalcium silicate.[33] Because the dicalcium silicate hydration rate is slower than that of tricalcium silicate.[32] and C-S-H from dicalcium silicate have a better crystallization and higher strength than that from tricalcium silicate, the C-S-H crystal of dicalcium silicate that forms later is more stable.

There are some disagreements on the reported compressive strength of WMTA and GMTA.[4, 65] Islam *et al*.[4] in 2006 found significantly higher compressive strength for GMTA at 3 and 28 days, compared with WMTA (table 3). On the other hand, two other investigations reported more compressive strength for WMTA.[65, 66]

TABLE 3 SUMMARY OF THE PHYSICAL PROPERTIES OF MTA CEMENTS.[4]

Materials	Radiopacity	Setting Time (Minutes)		Dimensional Change (%)	Compressive Strength (MPa)	
		initial	Final		3 days	28 days
GMTA	6.47	70 ± 2.58*	175 ± 2.55	0.28 ± 0.09	50.43 ± 1.30	98.62 ±5.74
WMTA	6.74	40 ± 2.94	140 ±2.58	0.30 ± 0.01	45.84± 1.32	86.02 ± 10.32
Portland cement	0.93	70 ± 2.16	170 ± 2.58	0.45 ± 0.09	48.06 ± 6.14	50.66±1.37

*Values are means±SD. (Adapted from Islam *et al*., 2006)

Considering factors that influence the compressive strength of MTA, Watts *et al*.[65] investigated the effect of pH and mixing agents on the compressive strength of WMTA and GMTA. They found a significant decrease in compressive strength ($p<0.0001$) when MTA was mixed with local anesthetic at acidic environment (pH 5.0). Holt *et al*.[66]found that mixing 2% chlorhexidine with either type of MTA lowered its compressive strength, even though its antimicrobial effect against *E.faecalis* was elevated. Another study revealed no statistically significant effect of the condensation pressures applied on MTA on compressive strength, even with the trend that too much pressure condensation produces lower surface hardness values.[67] On the other hand, another recent study found that mechanical mixing (MTA encapsulation) enhanced the compressive strength of MTA compared with manual mixing.[68]

Although ProRoot® MTA and MTA Angelus have similar constituents, the lower compressive strength values of MTA Angelus was due to different particle size. MTA

Angelus particles had relatively low circularity and wide size distribution and were less homogeneous than ProRoot® MTA. The present results revealed that the compressive strength of MTA Angelus was greater after mechanical mixing.[68]

TABLE 4 FACTORS AFFECTING THE COMPRESSIVE STRENGTH OF MTA.

Factors	Authors	Results
Type of MTA	Basturk *et al*.2013[68]	ProRoot MTA > MTA Angelus
pH and mixing agents	Watts *et al.2007*[65]	Acidic pH reduced compressive strength.
mixed with 2% chlorhexidine	Chng *et al*.2005[49] Holt *et al*.*2007*[66]	Reduced in compressive strength
Condensation pressures	Nekoofar *et al*.2007[67]	No significant effect
Additve with 1%methylcellulose and 2% calcium chloride	Ber *et al*.[69]2007	1.better in handling characteristics, 2. an approximately equal in compressive strength to MTA 3. set faster (57 ± 3 minutes).
Mixing & Placement technique	Basturk *et al*.2013[68]	1.Mechainical > manual mixing 2.Ultrasonic agitation >manual placement

There are common problems associated with the clinical applcation of MTA. Some disadvantages with using MTA are its extended setting time and difficult handling[33, 52]. These aspects still need further studies to overcome these flaws.

2. Glass ionomer cement (GIC)

Glass polyalkenoate or Glass-ionomer cement (GIC) was developed in 1972 by Wilson and Kent as polyacrylic acid-based cements, in the Laboratory of Government Chemist, London, United Kingdom.[16, 70] Firstly, glass ionomer cements were developed in order to combine the good characteristics of the silicate cements (aesthetic translucency and fluoride release) and the polycarboxylate cements (ability to chemically bond to tooth structure and biocompatibility to the pulp). Figure1 shows that GICs are developed by combining glass powder which is fluoroalumino-silicate glass, and aqueous solution of polyacrylic acid.

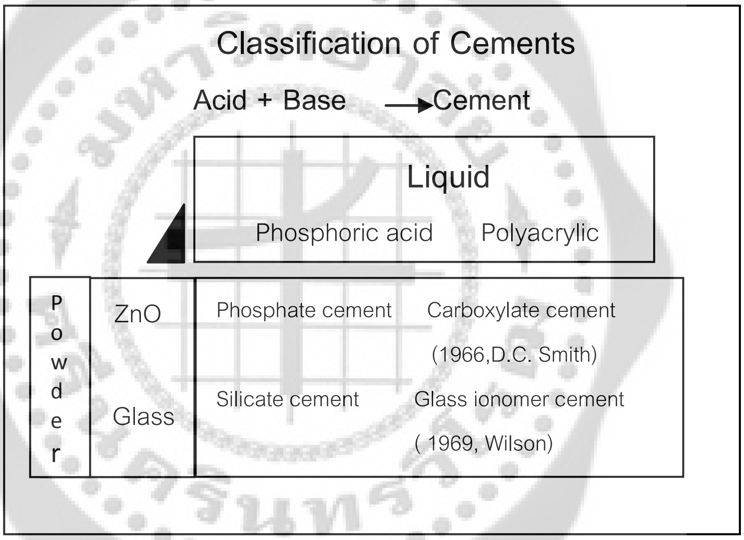

FIGURE1 CLASSIFICATION OF CEMENT (3M ESPE in Technical Product Profile)

GIC became popular in dental community due to its unique properties. It can chemically bond to the apatite in tooth structures and to the base metal through an ion-exchange reaction. It has cariostatic effect due to long-term fluoride release without high solubility.[71] It is thermally compatible with tooth enamel and dentin due to its low coefficient of thermal expansion similar to that of tooth structure, resulting in low shrinkage and thus minimized microleakage at the tooth-enamel junction.[72] Moreover, by combining weak acid and salt, GIC creates a chemical buffering effect which is an ability to increase pH when come into contact with acidic environment. This buffering effect also considered to be one of the important anticariogenic properties under clinical conditions.[73, 74]

Since then, glass ionomer cements have been used in several dental cares, especially in the conservative treatment that preserve tooth structure. These include root canal treatments, restorations of primary teeth , restorations in geriatric patients and class III and class V restorations. However, since GIC has low mechanical strengths, they are unsuitable for high-stress sites, like class I and II restorations.[72] Even though GIC has higher compressive and tensile strengths than those of zinc phosphate cement, their elastic modulus is only half of zinc phosphate cements'. Moreover, conventional GICs possess many disadvantages including technique-sensitivity, brittleness, low fracture toughness, low flexure strength, and low wear resistance.[18]

2.1 Composition of GIC.

GIC is formed from the reaction between an aqueous solution, polyacrylic acid, and acid-soluble glass powder, calcium fluoro-aluminosilicate.

The conventional glass powder is prepared by melting SiO_2, Al_2O_3, AlF_3, CaF_2, NaF, and $AlPO_4$ at temperatures ranging from 1,200 to 1,550 °C. After the melting process, the homogeneous glass is ground and sieved into a powder having particle size range from 15-50 µm. Recent development of GIC can make the particle size smaller, down to 2 µm, depending upon the clinical applications. The main structure that forms skeleton bone of the glass is alumina (Al_2O_3) and silica (SiO_2) (Table5). The inclusion of fluoride in the composition of GIC is clinically beneficial because the released fluoride ion can be readily exchanged for the hydroxyl ion of hydroxyapatite, making it resistant to caries.[75]

The original poly-acrylic acid solution is very viscous and concentrated. With its concentration of about 40-50%, this aqueous solution tends to become gel easily. To solve this problem, copolymers are used instead of homopolymers.[76] Recently, liquid solution contains polyacrylic acid in the form of copolymer with itaconic acid, maleic acid and tricarballylic acids. This copolymer of acids are used not only to prevent gelation but also increase reactivity (Figure 2.), because of an increase in the number of carboxylic groups (COO^-) per chain unit, and the higher acidity of the solution.

Setting rate of glass ionomer cement was slow at early stage. Wilson *et al*.[77] reported that modification with tartaric acid increased the setting rate, the compressive strength, and the tensile strength of the material, while the working time remained unaffected.

Water is very crucial for GIC liquid. It plays an important part in the acid- base reaction. It acts as the medium for reaction and is part of the reaction products after hydration. The amount of water presented in the composition of GIC is very critical. Mishandling water during the operating procedures will impact the properties of the cement. Too much water causes cements to become weak. On the other hand, too little water is insufficient to complete the setting reaction. Therefore, water is required at a proper proportion for the perfect setting of GIC.[76]

TABLE 5 COMPOSITION OF GLASS IONOMER CEMENTS.[70, 78]

Powder(Calcium fluoroaluminosilicate glass)			Liquid:	
Traces		%by wt	Traces	%by wt
Silica (quartz)	SiO_2	29.0%	Polyacrylic acid	35-50%
Alumina	Al_2O_3	16.6%	Itaconic acid	
Aluminium fluoride	AlF_3	5.3%	Maleic acid	
Calcium fluoride(fluorite)	CaF_2	34.2%	Tricarballylic acid	
Sodiumfluoride(cryolite)	Na_3AlF_6	5.0%	Tartaric acid	5-15%
Aluminium phosphate	$AlPO_4$	9.9%	Water	
Lanthanum,Strontium,barium (for radiopacity)		In trace		

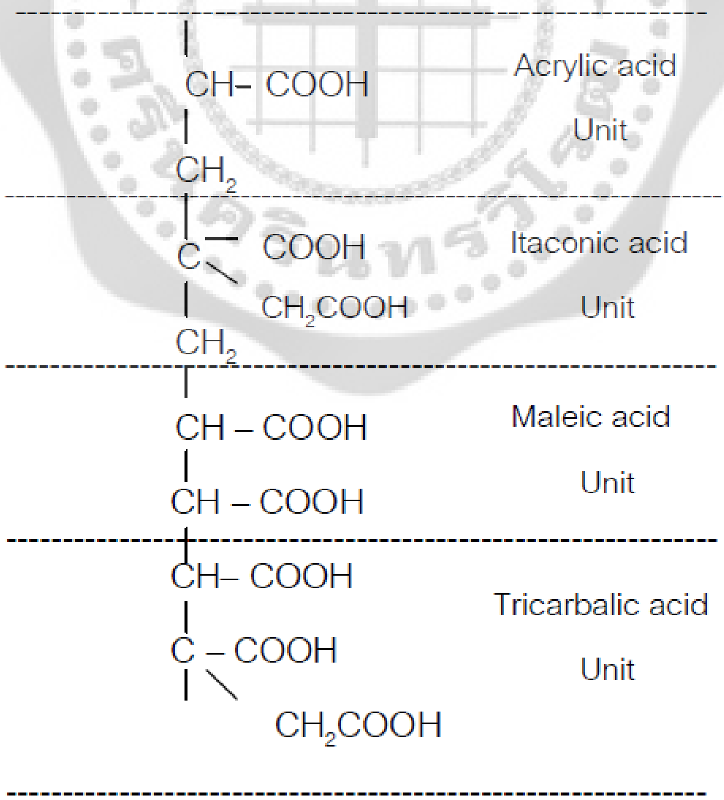

FIGURE 2 TYPES OF CARBOXYLIC ACID UNIT USED IN GIC LIQUID[79]

TABLE 6 CLASSIFICATION OF GICs ACCORDING TO APPLICATIONS AND SOME PHYSICAL PROPERTIES.[80]

Physical properties	**Type I** **Luting**	**Type II** **Restorative**	**Type III** **Lining or Base**	**Type IV** **core build up**
Use	Cementation of crowns & bridges	II.1 Class III & class V(aesthetic) II.2 Restorative reinforced	Thermal insulator under restorations	Crown & core build up (Silver containing GICs)
Powder:liquid ratio	1.5:1	3:1 or greater (7:1)	Lining: 1.5:1 Base:3:1or greater	3:1 or greater
Setting rate	Fast set	Autocure-slow set in early stage Resin modified: Fast set immediate after light curing	Fast set Resin modified: Fast set immediate after light curing	Fast set
Film thickness	≤20µm	-		-
Trade name (example)	Ketac™ Cem	II.1Fuji II LC, Gem-Fil II.2Ketac™-Molar, Fuji IX GC	Vitremer(resin modified)	Cermet, Ketac Silver, Espe GMbH, Germany

2.2 The Setting Reaction of GIC[81]

Setting reaction of cement is significant for the development of glass material for their application. Reaction involved is acid-base/neutralization reaction where glass being a base in sense that it accepts protons from acid even though it is not soluble in water. The setting reaction of GICs consisted of three overlapping stages: dissolution, gelation, and maturation phases. (Figure3.)

In the dissolution phase, when glass powder mixes with water, acid degrades the network structure and releases metal ions (Ca^{2+} and Al^{3+}) from outer layer of filler particles.[82] The Ca^{2+} concentration of the cement sol rises faster than the concentration of Al^{3+}.This is because of its larger ionic radius of Al^{3+}(5.0nm) and its larger trivalent charges, resulting in a lower diffusion rate of aluminum ions ($Al^{3+)}$ than calcium ions (Ca^{2+}) through the cement sol.

The pH of cement is around 2.6 when it is freshly mixed. After that, the pH rises rapidly during the dissolution phase.

In the gelation phase, a gel coat is formed on the outer surface of the glass particles due to a partial dissolution of Al^{3+}, Ca^{2+} and F^- ions. [83] These released cations (Al^{3+}, Ca^{2+}) are then chelated by carboxyl groups (COO^-) and crosslinked with polyacrylic chain. The cross-linking of GIC components, by COO^- groups from acidic solution and the Al^{3+} released from glass powder, creates a solid network around the glass particles. Furthermore, the COO^- groups also react with Ca^{2+} ions from the enamel, chemically bonding cement and the tooth structure together. Simultaneously, fluoride ions (F^-), released from the glass, is taken up by that adjacent enamel to help remineralisation of hydroxyapatite crystals. [84] Recently, it was reported that glass ionomer cements can act as fluoride reservoir and prevent a tooth from secondary caries as GICs can uptake fluoride from fluoride solution.[85]

However, GICs also consists of unreacted glass particle in that complex matrix which include calcium and aluminium polyacrylates in the form of inorganics network. This network has been suggested to be responsible for maturation process that will lead to the increasing of compressive strength and binding water into the structure.

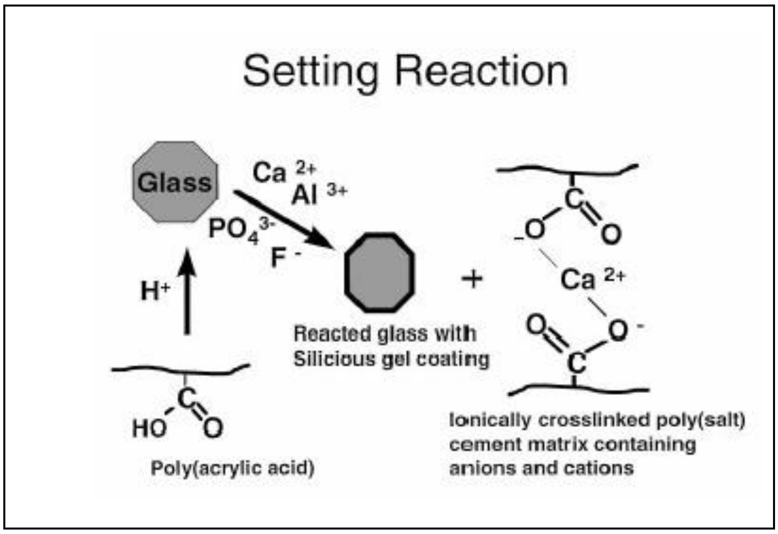

FIGURE3 SETTING REACTION OF GLASS IONOMER CEMENTS

(3M ESPE in Technical Product Profile)

In the hardening/maturation phase, the cement matures over the period of 24 hours and beyond. The cement has progressively more cross-linking of hydrated Al^{3+}. The set cement becomes less sensitive to moisture, and the percentage of bound water increases, as well as the glass transition temperature. Gradual reconstruction in the cement matrix is leading to the increase of compressive strength which arises gradually over some period of time to a maximum value as explained by Wilson and McLean.[75] Glass ionomer typically can reach a compressive strength of 180-220 MPa at one day and may rise over time. Translucency is also changed and become more like natural tooth material as a result of maturing.

The sealing ability of GIC, root-end filling material, was adversely affected by the moisture contamination from the root end cavities where it is applied. The light cure, resin reinforced GIC, used by Chong *et al*. as a retrograde filling material [86] showed less microleakage , due to its less sensitivity to moisture. It also has less curing shrinkage, and deeper penetration into dentin surface. However, later studies found it caused more adverse histological reaction to the periapical tissue than conventional GICs (Ketac™ Molar and Fuji IX® GP).[20]

2.3 Factors affecting setting reaction of GICs

2.3.1Glass composition: Glass composition is the major factor that influences the setting reaction. Al_2O_3/SiO_2 ratio and fluoride content; by increasing in this ratio-faster is the set, shorter is the working time. Fluoride content is also prolong the working time. A glass with finer particle sizes will set faster and have shorter working time; the time in which cement structure can be molded without imposing any damage.(87)

2.3.2 Temperature

Increase in temperature will speed up the setting reaction significantly.(83, 88) In other words, the cold temperature can increase the working time, but does not affect the material's physical properties. An example is the use of cold mixing slab.

2.3.3 Powder:Liquid ratio

By reducing the proportion of aqueous polymer solution in GICs mixture, the setting time might decrease, but the set cement will increase compressive strength. (89) Polyacid chain in aqueous solution is very important for the formation of cement. The molecular weights of the polyacid affect the setting rate, the toughness, the fracture toughness, and the wear and erosion resistance. The higher the molecular weight of polyacid chain is, the better properties of the cement are. However, high molecular weight means high viscosity, rendering it impractical in reality.(75, 90)

2.4 Factors affecting compressive strength of GICs

2.4.1 Cement with high strength could be obtained by mixing fluoride in an appropriate amount with a high Al_2O_3/SiO_2 ratio.(91)

2.4.2 A smaller filler grain size such microsize gives the higher hardness than the coarses.(91) Besides, it also improves the dimensional stability due to the lower expansion. Fine grain size yielded expansion in the interval 0-0.1% compared with coarse grain size with 0.1-0.2% interval after 4 months.

2.4.3 Spherical shaped powder was introduced to promote mixing and handling characteristics of cement. Moreover, it can increase the compressive strength of the GICs. (92)

2.4.4 The presence of fluoride in glass powder: Cements that contain fluoride can have compressive strengths well above 200 MPa at 24 hours, whereas the compressive strengths of fluoride-free cements are rarely 100 MPa. This might be due to the fact that aluminum binds strongly to fluoride to form complexe of the type AlF_2. [94]

2.4.5 The influence of storage medium: An exposure of aqueous environment to GICs has relatively deleterious effect on their mechanical properties. It disturbs the activity of Al^{3+} ion, which play a major role in cross-linking polymerization of the polyacrylic acid.[93] The first 24 hours after mixing is the most critical moment to GIC setting, as shown by de Gee *et al.*[94] and van Duinen *et al.* [95] During this period, the material is most prone to wear and dissolution. Consistently, another study found that curing GICs in oil yielded a significantly higher compressive strength compared to water storage.[83]

3.Ketac™ molar

Ketac™ molar (3M ESPE ,St Paul, MN USA) introduced in the mid 1990 was a subset of conventional GICs. It was also known as the "Condensible" or " Viscous" GICs. Ketac™ Molar (3M ESPE Dental Products, Seefeld, Germany) is characterized by having smaller glass particles (2.8-9.6 µm) and a higher powder : liquid ratio (>3:1). Improvement was made in order to achieve higher strength, greater wear resistance, and greater flexural strength than traditional GICs.[96] Xie *et al.*[72] compared the physical properties of 10 commercial GICs and found that Ketac**™** Molar had the highest compressive strength among any other type of GICs. Ketac**™** Molar has many uses in endodontics, including as a long-term temporary filling and root-end filling material.

TABLE 7 SUMMARY OF PHYSICAL & MECHANICAL PROPERTIES OF SOME POSTERIOR RESTORATIVE CONVENTIONAL GICs[72, 83, 96]

Material	Time(Minutes)		Compressive strength(MPa)		
	Working (incl.mixing)	Setting (from begin of mix)	1hour	24hours	7 days
Ketac™ Molar	3	5	150±16[96]	244±9[96]	301.3±10.1[72]
Easymix	2.63±3.4s[83]	5.71±16.6s[83]	71.1±10.9[83]	112.3±19.1[83]	118.2±14.2[83]
Fuji IX GC	2.5	6	136±12[96]	236±28[96]	-
	2.31 ±7.1s[83]	4.57±10.5s[83]	102.1±8.4[83]	168.4±14.5[83]	182.5±35.4[83]

*Values are mean±SD.(from product information, otherwise references)

Biocompatibility studies [97, 98] exhibited an evidence of initial cytotoxicity from freshly prepared samples, that decreased gradually with setting reactions. The inflammatory response was eventually subsided over an extended period. Callis and Santini [97]found that periapical tissues can better tolerate glass ionomer, compared with gutta percha/ sealer. Moreover, small fragments of GIC in bone did not obstruct the healing process. Costa *et al*.[20] found that conventional GIC (Ketac™ Molar) was the least cytotoxic material among other types of GICs. The resin-modified GIC (Vitremer and Vitrebond) was more cytotoxic. A study of cytotoxicity of various materials in cell culture by Osorio *et al.* [99]showed that MTA was not toxic to cells, whereas Ketac Silver (GIC), Super-EBA and amalgam exhibited higher levels of cytotoxicity. Silver contained in Ketac Silver may be the source of higher cytotoxicity. Vajrabhaya *et al*.[19] found Ketac molar is more cytotoxic to human PDL cell than MTA in cell cultured technique. Overall, studies suggested that Ketac™ Molar serves as the most biocompatible and comparative strong material among other types of GICs.[20, 72, 97, 100]

4. Biomaterials Monocalcium silicate(CS) / Wollastonite(W)

Over the past decades, several attempts were made on designing extracellular matrix scaffolds to culture new bone from various biomaterials of biological or synthetic. [101, 102] In the most common approach, a biomaterial scaffold serves as a temporary structure for cells to grow, proliferate and differentiate into certain cells types of tissue or organs of interest. [102] One significant characteristic of this bioactive material is its ability to integrate with living bone by forming a hydroxyapatite(HA) interface layer with living bone. [13] Apart from this, a series of biomaterial properties needs to be met if it is intended for clinical use. These properties include physical and chemical properties, bioactivity, osteoconduction, osteoinduction, biocompatibility and biodegradation.[102, 103]

Among various bioactive ceramics developed over the past three decades, the main ones commonly used in clinical practice are: the Bioglass® , sintered hydroxyapatite (HA) $(Ca_{10}(PO_4)_6(OH)_2$, sintered β-tricalcium phosphate (TCP) $(Ca_3(PO_4)_2$, HA/TCP bi-phase ceramic , and glass ceramic A-W (Apatite-Wollastonite) and β-wollastonite (CaO . SiO_2) in an MgO–

$CaO–SiO_2$ glassy matrix. [12] The most widely used synthetic calcium phosphate ceramics is Hydroxyapatite(HA), which has similar calcium-to-phosphorus ratio to that of natural bone and teeth.[104] Recently, Wollastonite(W)/monocalciumsilicate($CaSiO_3$) has been advocated as a potential substitute for hard tissue, because of its superior bioactivity to Hydroxyapatite(HA).[10] The bioactivity index of W glass is 3.2, while hydroxyapatite (HA) has bioactivity index of 3.0.[14] W glass also has greater surface reactivity than that of HA crystals, resulting in less time it takes to bond with bone.[9] This feature can be attributed to the silicon present in W glass. Silicon involves in the metabolic events that in turn induce the formation of new bone.[104]

Although Pseudowollastonite/monocalcium silicate($CaSiO_3$) is one of the most commonly used biomaterials for bone tissue regeneration[12], its major drawback lies in its relatively fast dissolution rate. It dissolves so fast that its mechanical strength is compromised. In addition, the increasing pH of the surrounding medium could affect the osteointegration of the monocalcium silicate with the natural bone.[104] Numerous studies have been shown that $CaSiO_3$ can be used for bone tissue engineering. Ni *et al*. in 2006 showed that novel bioactive porous α-$CaSiO_3$ scaffold can provided cell proliferation and differentiation of osteoblast-like cell.[10]

5.GICs – Bioactive Glass

Currently, many researchers have tried to improve the bioactivity and together with the mechanical strength of GIC by adding of several bioactive ceramics. Hydroxyapatite (HA) seems to gain most popularity among these bioactive glasses.[23, 105, 106] The reasons are it has the same compositions and crystal structure as the human dental organs. By Yap *et al.* [105]in 2002, nano-HA was shown to be a promising additive to glass ionomer Powder. A higher compressive and diametral tensile strength was reported for Fuji IX glass ionomer with 4 wt.% HA in its composition (CS = 177.27 MPa, DTS = 13.94 MPa), compared to the non-reinforced commercial Fuji IX (CS = 135 MPa, DTS = 12.07 MPa). In 2003, Lucas *et al.* [106] reported an increase in the fracture toughness when HA particles were added to the conventional GICs (Fuji IX GP®). The ultimate result of this was the maintained long-term bond of cement to dentin. Furthermore, the addition of HA did not impede the continuous release of fluoride. In 2011, Arita *et al*.[22]added hydroxyapatite (HA) particles into conventional restorative GIC (Fuji IX GP®)

and found an enhancement in both the flexural strength and fluoride ion releasing ability of Fuji IX GP®. However, there has been little study conducted so far, to pursue this matter. Still, the use of GICs added with HA as root-end filling materials is promising and further evaluations are bound to come.

Other bioactive particles such as bioactive glass (BAG) and fluoroapatite also have been used in the dental research. In a recent study, Moshaverinia *et al*. in 2008[23] have added nanoparticles (100–200 nm) of both HA and Fluoroapatite (FA) to glass ionomer powder (5 wt.%). Results from the mechanical tests showed higher strength in both glass powders. [23] In 2005, Yli-Urpo *et al*.[21] investigated the effect of addition of BAG particles to conventional and resin-modified GIC powders. They concluded that the biocompatibility of the test specimens improved but the compressive strength decreased with an increasing amount of BAG.

The addition of bioactive materials into GICs appeared to be a promising approach in improving GICs in both biocompatibility and mechanical aspects. Up to date, no study investigates the effect of adding of monocalciun silicate(CS) /pseudo-wollastonite (ps-W) into conventional GICs (Ketac Molar) to its physical and bioactive properties. Therefore, the purpose of this study was to investigate the physical properties, in terms of setting time, pH and compressive strength, of GIC-CS compared with conventional Ketac™ Molar and ProRoot® MTA.

CHAPTER 3

MATERIALS AND METHODS

Preparation of pseudowollastonite (ps-W)

Pseudo-wollastonite (ps-W) was prepared by co-precipitation method as donated by Asst.Prof.Dr. Punnama Siriphannon[11, 107] from King Mongkut's Institute of Technology Ladkrabang. Briefly, $CaSiO_3$ powders were obtained by co-precipitation method using NaOH as the precipitant. The starting materials, $Ca(NO_3)_2$. $4H_2O$ and $Si(OC_2H_5)_4$(TEOS), were dissolved in 500 mL of ethanol and stirred for 2 h. Then their concentrations were adjusted to 0.2 mol/L. After that, 300 mL of 0.33 mol/L NaOH was added to the solution and a precipitate of $CaSiO_3$ could be obtained. The precipitate was filtered, washed with distilled water, and oven-dried at 100°C overnight. The dried powder was calcined at 500° and 900°C to crystallize the β-$CaSiO_3$ phase. The chemical composition of the calcined powder was analyzed by X-ray fluorescence (XRF; RIX3000, Rigaku Co., Tokyo, Japan).

Preparation of the composites glass/cements (Table 8,9)

GIC(Ketac™Molar EasyMix)

To prepare GIC cement, Ketac™Molar Easymix was manually mixed with the polyacrylic-tartaric acid, using plastic spatula on a mixing paper, at powder:liquid ratio of 3:1(mg/mL).

GIC-CS compound

To prepare GIC-CS compound, CS powder 10%, 30% and 50% by weight was uniformly mixed with GIC powder and then the compound was manually mixed with the polyacrylic-tartaric acid, using plastic spatula, at powder:liquid ratio of 3:1(mg/mL) for Ketac™ Molar and 2.5:1 (mg/mL) for CS.

MTA

To prepare ProRoot® MTA cement , ProRoot MTA powder was dissolved in the supplied deionized water at the powder:liquid ratio of 3:1or 0.33 mL of water per 1 gram of powder on a glass slab with a stainless steel spatula.[28]

TABLE 8 PRODUCTS USED IN THE EXPERIMENT

Products	Manufacturers	LOT
Ketac™ Molar	3M ESPE	Powder: 486812,487238 Liquid: 481304,485801
ProRoot® MTA	Dentsply Tulsa Dental	11004374, 12001879

TABLE 9 COMPOSITION OF COMPOSITES GLASS PREPARED FOR EXPERIMENT

Groups	CS powder (% wt)	Ketac Molar powder (% wt)	MTA (% wt)	L/P (mL/g)
1.GIC	-	100	-	0.33
2. CS10	10	90	-	0.33 for GIC 0.4 for CS
3. CS30	30	70		
4. CS50	50	50		
5. MTA	-	-	100	0.33

Setting time measurement

The Gilmore apparatus and cylindrical stainless steel molds (2.0 mm height with a 10.0 mm diameter) were used in this study by the recommendation of the ISO 6876:2001[4, 108] and ANSI/ADA Spec No. 57. The experimental and control materials were mixed with different concentrations of ps-W and liquid Ketac Molar for 40 seconds at room temperature (23°C ± 1°C). The initial and final setting time were measured with two different Gilmore-type needles. For the initial setting time, the Gilmore-type needle used weighs 100±0.5g and has a flat end of 2.0±0.1 mm in diameter. Another Gilmore-type needle used in the final setting time measurement weighs 400±0.5g and has a flat end of 1.0±0.1mm in diameter. The methodology was according to Bortoluzzi *et al*. in 2009.[109] Pilot studies have been made to determine the estimated setting time. The needle tips were indented on the cements at 1 minute before the estimated setting times. Then, the indenter tip was repeated , determining the setting time, every 15 seconds until no indentation could be seen. All tests to determine the setting times of the materials were done in a temperature- and humidity-controlled chamber (37°C°±1°C and 95% relative humidity) (Medical & Environmental Equipment Research Laboratory, Bangkok, Thailand) (Figure4.). Six samples of material in each group were tested.

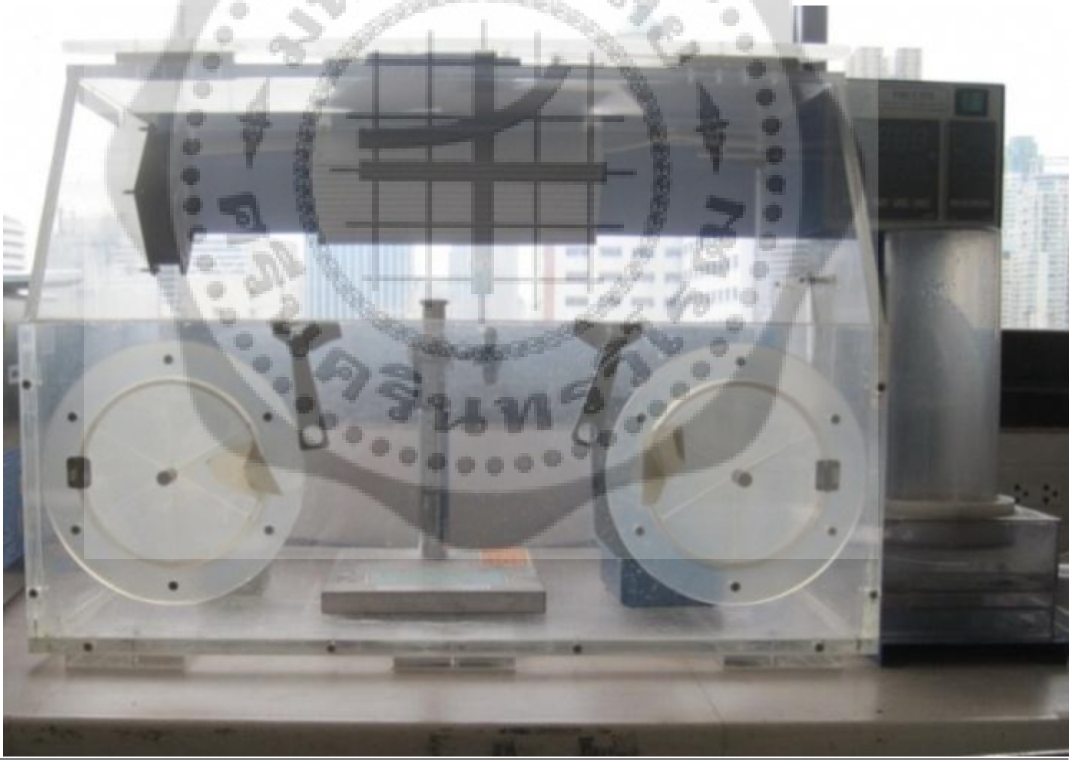

FIGURE 4 A TEMPERATURE- AND HUMIDITY-CONTROLLED (37°±1°C and 95% relative humidity)

FIGURE5 A.Cement setting time determining an apparatus. (with a weight of 100±0.5g)

B.Gilmore-type needle for determining of initial setting time (a flat end of 2.0±0.1 mm in diameter)

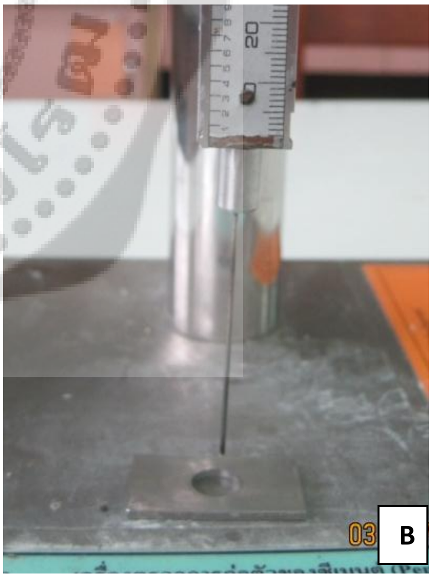

FIGURE6. A Cement setting time determining apparatus.(with a weight of 400±0.5g)

B.Gilmore-type needle for determining of a final settimg time (a flat end of 1.0±0.1mm in diameter)

Measurement of pH

According to Morgental *et al.*[110], each crushed material was suspended in deionized water at the concentration of 50 mg/ mL. The suspension was then vortexed for 30 seconds, and centrifuged for another 30 seconds, until the supernatant became clear. After that, the supernatant of each cement suspension was measured for pH with a pH meter (Digimed, Sâo Paulo,SP, Brazil), previously calibrated at room temperature (25 °C). The pH values were evaluated periodically every 10 min for 60 min, and every hour until 6 hours, 24 , and 48 hours after the preparation of suspension. Deionized water was used as a negative control. All experiments were performed in triplicate. The pH values of each material were averaged and the associated standard deviations were calculated.

Compressive strength

The compressive strengths of test materials were determined by the method recommended by the ISO 9917-1: 2007 specification for dentistry-water-based cement.[111, 112] Before testing, the instruments and the test materials were conditioned at 23 ±1°C in the laboratory for 1 hour. Six samples of each cement type was mixed and placed in split stainless steel molds. (Cylindrical specimens were 4 ± 0.1 mm in diameter and 6 ± 0.1 mm high). Each mold was packed in excess, with a slight pressure applied to the materials when placed. After removing any extruded material, the glass plates were gently compressed on the mold. The whole assembly was kept in an incubator for 1 hour at 37 ±1 °C. The specimens were ground with wet 400-grit silicon carbide paper before the specimens were removed from the molds. After examining for voids and chipped edges, six acceptable samples were collected for testing while the rest were discarded. The specimens were immersed in distilled water for 1 day, 3days, 7days, 21days, and 28 days after mixing before their compressive strengths are measured. The samples were tested at five different time intervals making a total 25 groups. Each group consisted of 6 samples, which makes a total of 150 samples.

The compressive strength was determined with a universal testing machine (Lloyd LRX, Lloyd Instruments, Fareham, UK) at a cross-head speed of 1.0mm/min. Each specimen was placed with its flat end between the platens of the testing apparatus. The load was applied in the long axis of the test sample. The maximum load required to fracture each specimen (Force at failure(P)) was used to calculate and the compressive strength (C) in Megapascals as the following formula

Compressive strength (MPa)= Force at failure(N) /Cross sectional area of specimen

$$C = 4P / \P D^2$$

where P was the applied force (N) and D was the diameter (mm) of the specimen. The compressive strength(C) of all specimens was recorded in MPa.

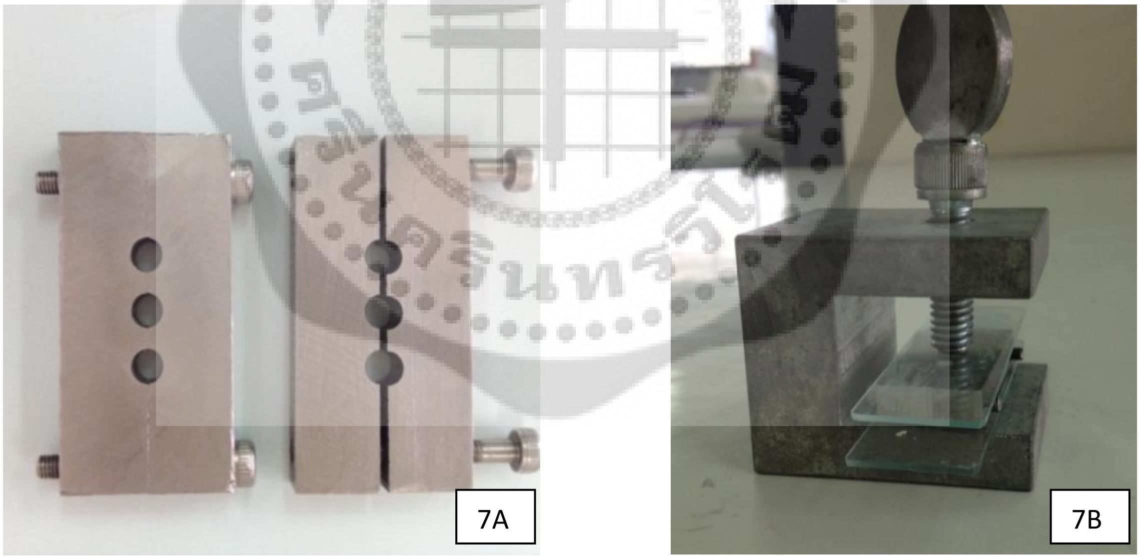

FIGURE 7 7A: SPLIT STAINLESS STEEL MOLD. (Cylindrical specimens 4 ± 0.1 mm in diameter and 6 ± 0.1 mm high), 7B: THE WHOLE ASSEMBLY (the mold is packed and compressed with glass plates on the mold).

FIGURE 8 UNIVERSAL TESTING MACHINE (Lloyd LRX) WITH MAXIMUM LOAD OF 10K NEWTON FOR COMPRESSIVE STRENGTH TEST

Statistical analysis

The mean values and standard deviations were recorded for all measurements. Statistical analyses were carried out for

Setting time

- To compare the setting time between GIC-CS groups and GIC group used one-way ANOVA(Analysis of variance) and scheffe post hoc at 0.05 level of significance.
- To compare the setting time between GIC-CS groups and MTA group used independent t-test.

pH and compressive strength using one-way ANOVA and scheffe post hoc at 0.05 level of significance.

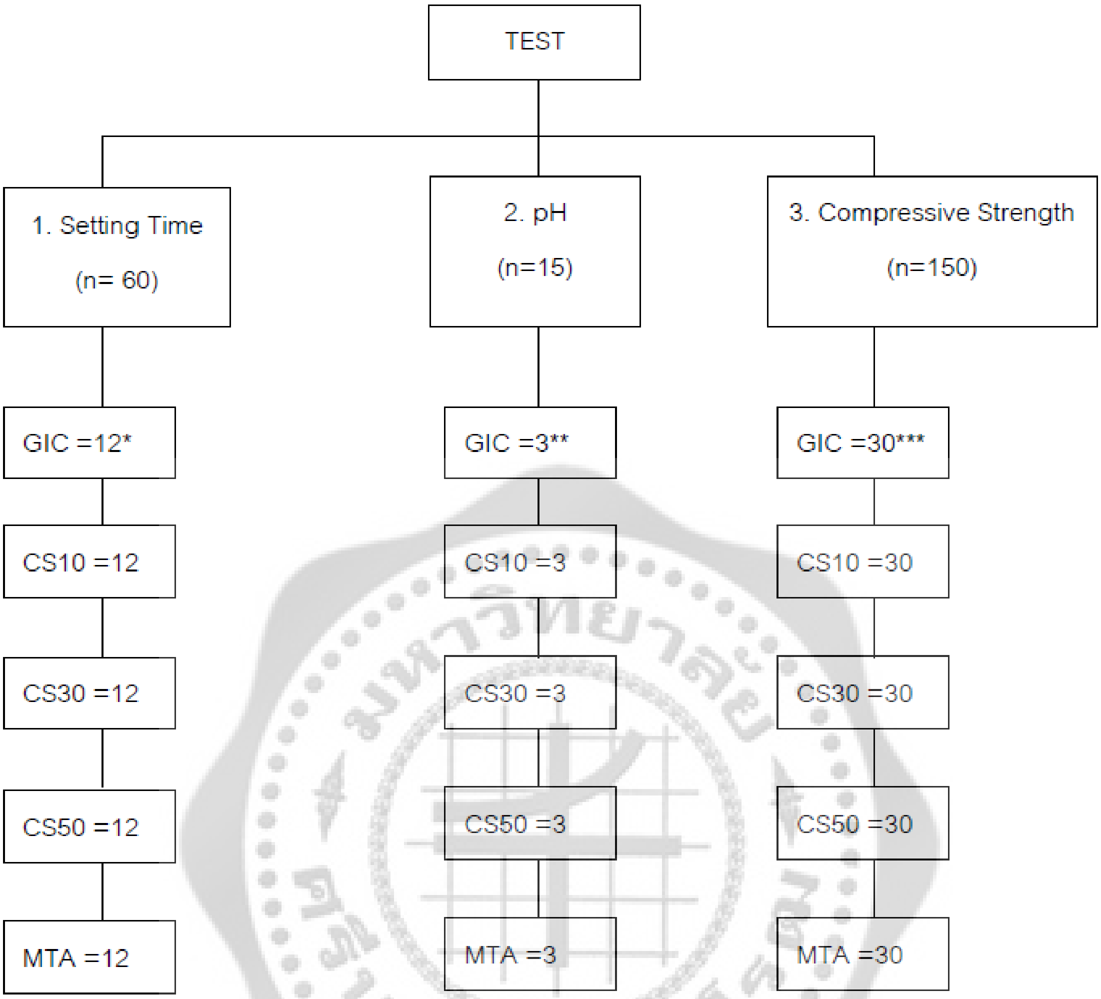

FIGURE 9 SUMMARY OF TEST & CONTROL SPECIMENS (GIC, GIC-CS, MTA)

**period of time tested : initial 6+ final 6, ** every 10 mins for 60 mins, every hour for 6 hours, 24 hours and 48 hours, ***6 specimens at each time period: 1,3,7,21 and 28 days*

CHAPTER 4

RESULTS

When powder and liquid of Ketac™molar were mixed with CS in various proportions (10%, 30%, 50%), the resultant GIC-CS compounds showed moldable consistency as close as Ketac™molar itself. The higher the ratio of CS added in the compounds, the more moldability of the new material was. This GIC-CS consistency allowed it to be packed in the mold by the plastic instrument.

Setting Time

Both initial and final setting times of all tested groups(CS10,CS30 and CS50) were significantly less than that of MTA($p<0.05$).(Figure 10) Within the GIC-CS compound groups, CS50 had longer setting time than both CS30 and CS10. Comparing between the GIC-CS groups and GIC; CS30 and CS50 groups had significantly prolonged the setting times of GIC. There were significant differences in the setting times between the test groups ($P < .05$) as shown in Table.10

TABLE 10 SETTING TIMES OF EXPERIMENTAL AND CONTROL CEMENTS.

(determined by Gilmore needle)

Cement types	Initial setting time		Final setting time	
	Mean	SD(sec)	Mean	SD(sec)
GIC	4 mins 25 sec [a]	12.25	5mins 30sec [a]	21.21
CS10	5 mins 53 sec [b]	35.52	7mins 25 sec [b]	33.76
CS30	6 mins 35 sec [b]	12.65	8 mins 22 sec [b]	20.67
CS50	8 mins 45 sec [c]	16.43	12 mins 52 sec [c]	35.18
MTA(control)	64 mins 0 sec	136.82	121mins 30 sec	159.87

The different superscript letters mean significant difference between the GIC, CS10, CS30, CS50 groups (one way ANOVA, $P < .05$) according to Scheffé *post hoc* multiple comparisons.

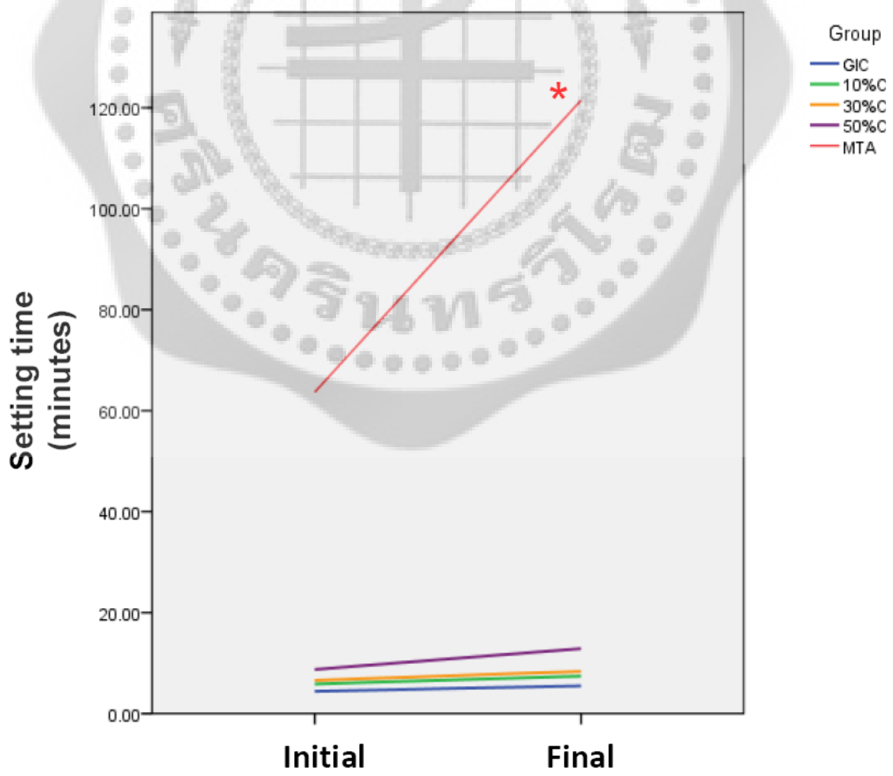

FIGURE 10 MEAN INITIAL AND FINAL SETTING TIMES OF THE MATERIALS.

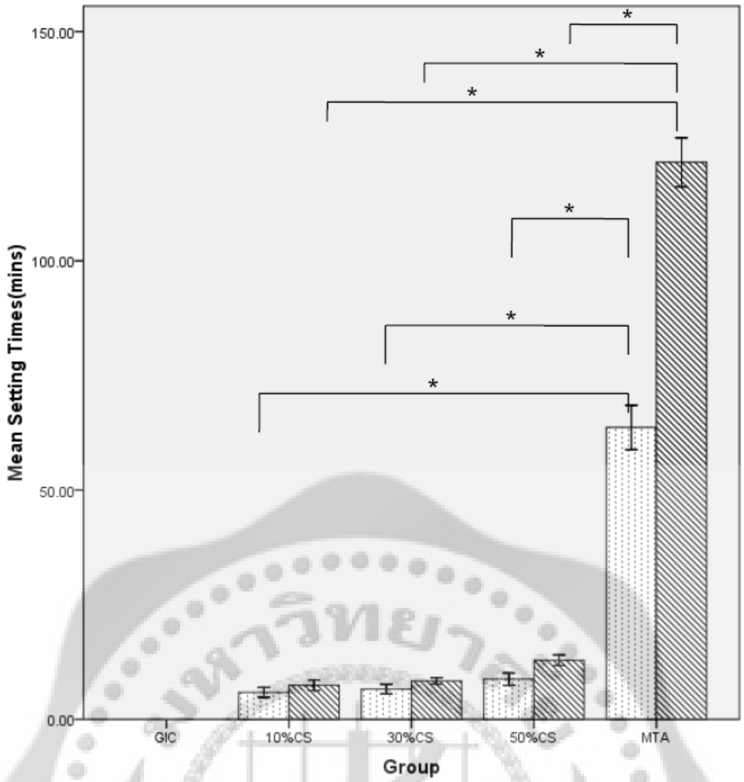

FIGURE 11 GRAPH SHOW INITIAL AND FINAL SETTING TIME OF DIFFERENT EXPERIMENTAL GIC-CS COMPOUNDS GROUP COMPARED WITH MTA. (independent t-test ,* means significant different $p<0.05$)

pH

The average pH of MTA , GIC-CS compounds and GIC were found to be constant after mixing throughout the test periods(10minutes to 48 hours) as shown in Figure12 . The pH value of MTA rose to approximately 12.12 after 10 minutes and remained alkaline until 48 hours of the test period. Differently, GIC and GIC-CS compounds appeared to be acidic throughout 48 hours periods. The mean pH of MTA was the highest among the tested groups ($p=0.00$), followed by CS50, CS30, CS10 and GIC respectively. There were significant difference in mean pH between each group ($p<0.05$).

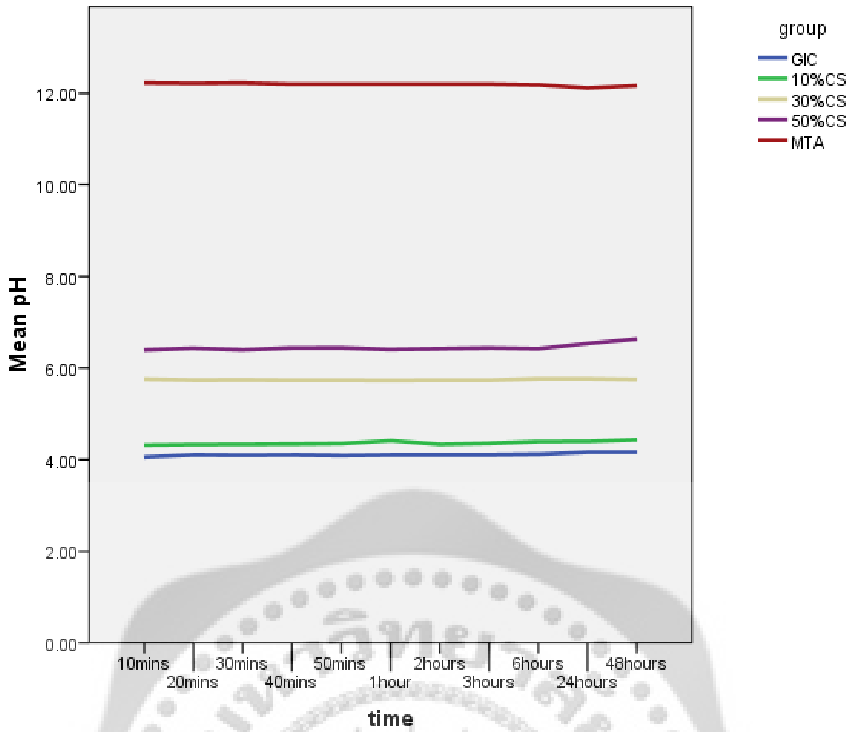

FIGURE 12 MEAN pH OF THE MATERIALS

TABLE 11 MEAN pH OF DIFFERENT CEMENT TYPES

Cement types	Mean pH	Standard deviation
GIC	4.106[a]	0.044
CS10	4.365[b]	0.135
CS30	5.744[c]	0.055
CS50	6.447[d]	0.136
MTA	12.12[e]	0.085

The different superscript letters mean significant difference between the groups ($P < .05$) according to Scheffé *post hoc* multiple comparisons

An increase in monocalcium silicate content from 10 to 50 wt % resulted in gradually increasing pH of GIC-CS compounds.

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Compressive Strength

According to one-way ANOVA and Scheffé post hoc multiple comparisons, there have significant different in compressive strengths of testing materials when compared at each time point (1day, 3days, 7days, 21days and 28 days). Considering at 1 day period, the compressive strength of MTA was significantly less than that of GIC and GIC-CS compounds ($P<0.05$). However, by day 3, MTA showed no significant difference in compressive strength compared to GIC and GIC-CS compounds ($P>0.05$). At day 21 and day 28, the compressive strength of CS50 cement group was significantly lower than that of other tested groups ($P<0.05$). There was no significant difference in compressive strength between the GIC ,CS10 and CS30 groups ($P>0.05$) at all time periods as shown in Table12.

TABLE12 COMPRESSIVE STRENGTH OF DIFFERENT CEMENT TYPES

Cement type	Compressive strength (MPa)				
	1day	3days	7days	21days	28days
GIC	66.72±9.80[a]	84.67±2.99[A]	103.85±7.13[*]	126.50±2.07[¶]	128.50±5.09[α]
CS10	61.45±4.66[a]	72.95±6.08[A]	101.33 ±1.94[*]	108.10±1.76[¶]	130.42±2.46[α]
CS30	77.73±2.61[a]	94.42±8.61[A]	102.43±8.84[*]	103.58±9.47[¶]	114.83±7.57[α]
CS50	86.67±8.61[a]	75.25±4.58[A]	96.68±6.26[*]	80.30±7.12[b]	80.96±4.70[β]
MTA	30.98±5.00[b]	66.43±10.33[A]	66.45±2.46[**]	114.83±8.13[¶]	122.33±7.23[α]

Values are reported as mean ± standard deviation. Considering at each time point , the different superscript letters in the same row mean significant difference between the groups ($P < .05$) according to Scheffé *post hoc* multiple comparisons

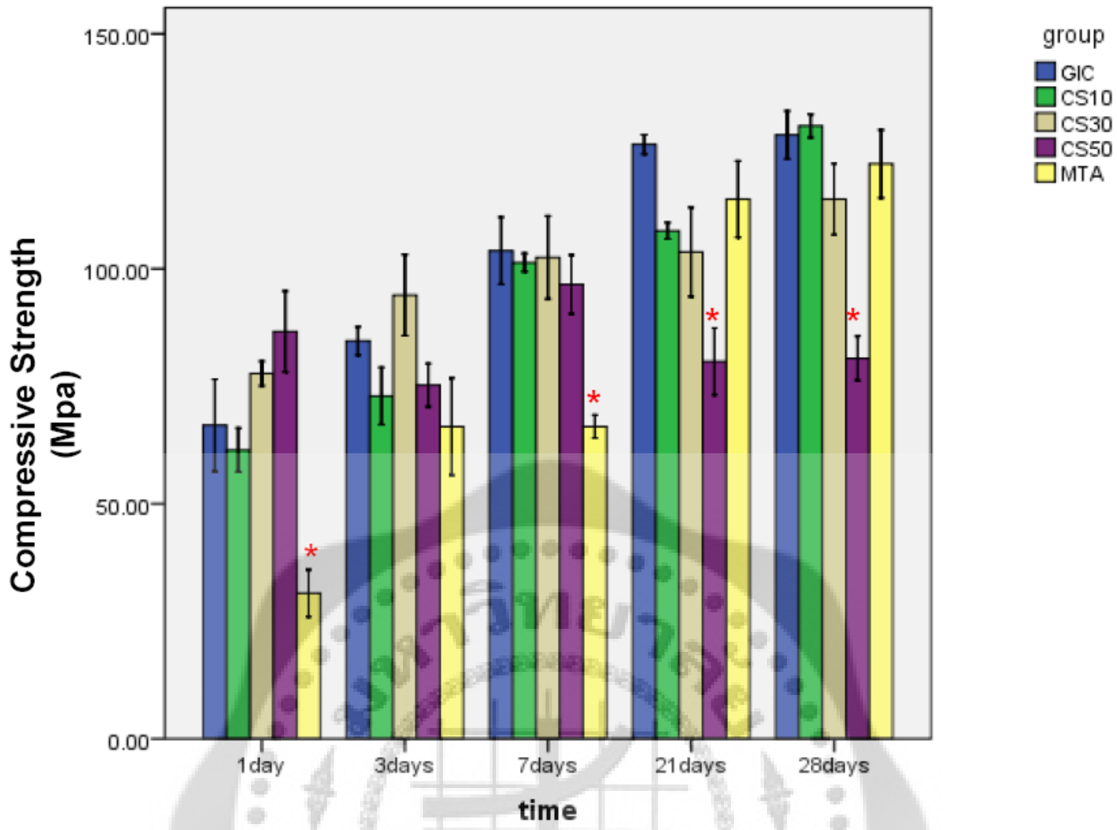

FIGURE 13 COMPRESSIVE STRENGTH OF DIFFERENT CEMENT TYPES.(n=6)

(*significant different between the groups (P < .05) at each time point according to Scheffé *post hoc* multiple comparisons)

Figure 13 showed the trend of compressive strength for most cement groups except for the CS50 one. The strength seemed to increase with times for GIC, CS10, CS30, and MTA. For CS50, a drop in compressive strength was seen by day 21 and was retained until day 28.

CHAPTER 5

DISCUSSION

GICs are set by the acid-base neutralization between ion-leachable fluoroalumino-silicate glass and polyalkenoic acids liquid. Even if GIC contains both calcium and phosphate, it does not show any bioactivity.[17] The acid-base setting reaction allowed an addition of monocalcium silicate to make the GIC-CS compounds with enhanced bioactivity. Since monocalcium silicate(CS) has shown a potential as bioactive material that can induce hard tissue formation,[11, 104] adding CS glass should enhance bioactivity of GIC. However, none of the studies were conducted using GIC-CS so far. This study intended to compare GIC-CS mixture in various ratios (10%CS, 30%CS, 50%CS) with MTA and conventional GIC (Ketac™Molar). This GIC-CS mixtures should possess a range of acceptable physical properties.

The purpose of this study was to develop a new endodontic material based on the physiochemical properties of GIC (Ketac™ Molar) and MTA, in an attempt to overcome the drawbacks of both materials. This study tested physical and mechanical properties of GIC-CS compounds on the setting times, pH, and compressive strength, with the main purpose to improve the prolonged setting time of MTA in clinical usage.

Physical and mechanical properties of cements vary due to a number of varibles such as the particle size, chemical composition, sintering temperatures of the powders, powder to liquid ratio, temperature, pH of the environment, and mixing method[113].For these reasons, pilot studies were performed to test the setting time of GIC-CS compounds in a close-ended mold design placed in an incubator (37 ±1 °C) for 1 hour, and then immersed in deionized water for 3 days . None of the specimens appeared to be washed out after 3 days, and all appeared to set.

The working time and setting time, are two of the most clinically relevant factors for the development of GIC-CS compounds. Since working and setting time of GIC are too short, it may set before suitable for endodontic use. On the other hand, MTA possesses too long setting time that may jeopardize the dimensional stability of the cement. The proper working time for endodontic treatment, though no standard was set, must be long enough for placement and adjustment of the cements. Considering that the mixing of glass powder and liquid solution took approximately 1 minute, the working time should be at least 4 minutes to allow for the transfer of the cement to the tooth cavity, Fernandez *et al*. [114] recommended an optimal setting time for 10-15 minutes. For Lewis, the suggested working time was 6-10 minutes, while the setting time was advised to be approximately 15 minutes for injectable bone cements using in vertebroplasty and kyphoplasty.[115]

The results showed that GIC-CS compounds exhibited shortened setting times both initial (4-9 minutes) and final (5-13minutes) as compared with the setting time of MTA (63-125 minutes)($p<0.05$). This advantage would be beneficial to reduce the risk of displacement and contamination during clinical procedures. As a root-end filling material, it should set as soon as it is placed and adapted in the cavity to allow cements to maintain shape of the restoration during surgical procedures. In addition, as a pulp capping material, shorter setting time allows proper placement of a filling material over it in order to create a bacterial tight seal environment. GIC-CS prolonged working (4.42 minutes) and setting time (5.5minutes) of GIC. This is useful for endodontic applications.[116] Based on these data, we can conclude that an increase in the amount of monocalcium silicate content retarded the setting time of the GIC-CS compounds. It has been suggested that gelation of poly-acrylic acid by Ca^{2+} ion occurs faster and easier than by Al^{3+} ion.[117] Increasing of monocalcium silicate in the GIC-CS compounds causes more Ca^{2+} ion in the acid-base reaction[15]which has an accelerating effect in the early setting step, but at the same time, the lower number of Al^{3+} ion has a role to retard the maturation stage of GIC-CS acid-base setting reaction. All of these effects together results in an extension of the setting time. Furthermore, the working time and setting time of GIC-CS were adversely proportional to Si/Ca molar ratio, in agreement with a previous study of calcium silicate cements.[15, 113]

Another reason for this prolonged setting time might due to the larger particle size of monocalcium silicate (≤63 μm) as compared to Ketac™ Molar(<10μm). The setting reaction would be faster if the particle sizes were smaller because they will increase the specific surface of the glass powder.[77] The effect of the particle size of the cement powder on the setting and hardening properties has been extensively studied in other hydraulic cements, such as Portland cement[118-120] and the similar findings were also obtained.

MTA was used as a comparison of our results with previous investigations. A number of investigations have been carried out to assess the physical and mechanical properties of MTA as a root-end filling material.[49] This study also found its setting time, pH and compressive strength in accordance with the findings reported in these earlier studies.[1, 4, 25, 50, 53, 121] For the setting times, Ber *et al*. [69] used a Vicat needle, reported a final setting time to be 202 minutes, while Kogan *et al*. [52] found the experimental setting time of MTA to be 50 minutes. Torabinejad *et al*. and Gandolfi *et al*., both using Gilmore needle, obtained similar final setting times to be 175 minutes[50] and 170±2 minutes[121], accordingly. For this present study, the results indicated that initial and final setting time of WMTA were 63.70±2.42 and 121.5±3 minutes respectively, which is similar times as reported by Bortoluzzi *et al*.[109] (38 and 190 minutes), even though they used a different brand of cement. The use of different needles that differ in weights (300 g Vicat versus 453.6 g Gilmore needle), humidity, temperatures and the amount of time the needle rests on the surface to produce the indentation may be responsible for the differences in setting times.[69, 119, 121] Moreover, determination of setting times is subjective. It is only an estimation of when a given probe fails to indent a sample. There is no universally accepted testing procedure. MTA's average strength (66.45±2.46 MPa at 3 days) conformed with previous observations using the same cement products(WMTA) with an average strength of 45.84± 1.32 MPa for 3 days[4].

GIC-CS compounds not only shorten setting time, but also possess a better handling characteristics when compared with MTA. One of the main clinical limitations of MTA is its difficulty in handling due to its granular consistency. The GIC-CS compounds have more cohesiveness and moldable characteristics, similar to Ketac™ Molar. This putty consistency would allow easy manipulation and adaptation of the material without any special instrument. However, GIC-CS compounds need to be further tested clinically to determine whether they do allow for easier placement and whether it significantly reduces cement washout.

Increasing monocalcium silicate content from 10 to 50 wt % tended to neutralize the pH of GIC. This neutral pH can indicate the neutralisation/acid-base reaction between the basic glass and polyacids.[91, 122] This implied that the addition of monocalcium silicate into GICs enhances the setting reaction of the compounds by depleting more acid and making the setting product more neutral. The pH values of the GICs negative control group were in agreement with previous observations: a pH of 2.0 at 5 minutes that rapidly rose to 3.0 at 10 minutes.[123] This increase in the pH of GIC-CS would be beneficial to the biocompatibility of GIC, due to its neutralization in toxicity of the strong acidity in the initial setting reaction periods which is one of the drawback of GICs.

The pH values of the MTA positive control groups were also in agreement with previous observations using the same cement products (WMTA), an overall average pH of 12 for 2 days compared with pH 10.2 after mixing and rose to pH 12.5 after 3 hours and there after.[4, 8, 25, 50, 109]

As root-end filling materials, compressive strength values of cements are not critical because they do not bear any direct force. However, its compressive strength is important if the material is to be used as a pulp capping material. In this case, compressive strength of the cements was measured to investigate the effects of cements additives, and not to determine whether the cements could withstand the loading force.[65, 124] Furthermore, compressive strength is commonly used in the cement industry to verify that cements are completely set.[65]

In the methods of testing compressive strength, voids in the prepared specimens appeared to be the weakest point of the specimens which can cause significant error to the test results. For this reason, specimens condensed carefully by hand plugger were chosen in this study. According to Torabinejad *et al*.[25], Aminoshariae *et al*.[124] and Watts *et al*[65], this method caused less void than another method; a combination of hand plugger with ultrasonic vibration.

CS10 and CS30 groups showed no statistically significant difference in compressive strength compared to GIC control group, while showing an improvement in setting time and compressive strength when compared with MTA in 24 hours period. On the other hand , CS50 group showed statistically significant reduce in the compressive strength, especially in the 21 and 28 days period. However, this reduction in strength did not seem to have clinically relevant difference. The reduction instead implied that larger amount of monocalcium silicate particles were not homogeneously incorporated into GIC-CS compounds. This was partially attributed to larger particle sizes of monocalcium silicate(~63µm) compared to fluoroaluminosilicate particles of Ketac™ Molar(2.8-9.6 µm).[72] Furthermore, GIC set product contained aluminum cross-linking in both polyacrylate and silicate networks (aluminum carboxylate salt and aluminum polyacrylate), which had a higher compressive strength[70] than hydroxyapatite of CS.

GIC control group exhibited low compressive strength (66.72±9.80 MPa) at 1 day period compared to previous studies(244±9 Mpa).[96] A possible reason was due to the deteriorating effect of water on the compressive strength of Ketac**™** Molar.[83, 94, 95] According to the setting reaction of GIC, the first 24 hours are very critical. The material in this period is most prone to wear and dissolution. However, the compressive strength of Ketac™ Molar in this study was in the same range as that reported by Algera *et al*.[83] in 2006 (83.8±11.6 2 MPa at 1 day period) and Camilleri *et al*.[63] in 2008 (47.9 ±16.2 MPa at 1 day period) who used the same method following ISO 9917 (2003)[112] specifications.

Furthermore, compressive strength of GIC-CS compounds tends to increase with time due to GICs' self setting reaction.[83] Another possible reason is the aluminium cross-linking between the polyalkenoic acid chains, formed by a slow final maturation reaction increases the strength of the GIC. This indicates that even when employed clinically and left in contact with tissue fluids, all the materials are likely to continue to set and gain strength and stability.[4]

Within the limitations of this study, GIC-CS compounds seem to be promising materials that are worth studying on other aspects. Further studies should focus on refining monocalcium silicate into smaller particle size to improve compressive strength and other physical properties of the GIC-CS compounds.

CHAPTER 6

CONCLUSION

The setting times of GIC-CS in all ratios (CS10, CS30, CS50), less than 13 minutes, were significantly lower than that of MTA (124 mins). Addition of CS to GIC significantly neutralize in the pH of GIC in the 48 hours period($p < 0.05$). The compressive strengths of CS10 and CS30 were not statistically significant different from that of GIC for all testing periods (28 days). CS50 group showed statistically reduction in GIC's strength ($p < 0.05$). However, this reduction was not shown to be clinically relevant. On the basis of these results, adding CS into GIC displayed an advantageous shorter setting time than MTA, neutralized the pH of GIC and not clinically affected to GIC's compressive strength at 28 days. GIC-CS may have the potential to be used as a root-end filling or pulp capping material.

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APPENDIX

Statistical Analysis

1.Setting Time testing

Table 1 Descriptive analysis for setting time

Descriptives

		N	Mean	Std. Deviation	Std. Error	95% Confidence Interval for Mean Lower Bound	95% Confidence Interval for Mean Upper Bound	Minimum	Maximum
Initial	GIC	6	265.0000	12.24745	5.00000	252.1471	277.8529	255.00	285.00
	10%CS	6	352.5000	32.51923	13.27592	318.3732	386.6268	315.00	390.00
	30%CS	6	395.0000	30.98387	12.64911	362.4844	427.5156	360.00	450.00
	50%CS	6	525.0000	40.24922	16.43168	482.7610	567.2390	450.00	570.00
	Total	24	384.3750	99.97078	20.40645	342.1610	426.5890	255.00	570.00
Final	GIC	6	330.0000	21.21320	8.66025	307.7381	352.2619	300.00	360.00
	10%CS	6	445.0000	33.76389	13.78405	409.5670	480.4330	390.00	480.00
	30%CS	6	502.5000	20.67607	8.44097	480.8018	524.1982	480.00	540.00
	50%CS	6	772.5000	35.17812	14.36141	735.5828	809.4172	720.00	810.00
	Total	24	512.5000	168.06443	34.30601	441.5326	583.4674	300.00	810.00

Table 2 Test of Homogeneity of Variances between GIC&GIC-CS for setting time testing

Test of Homogeneity of Variances

	Levene Statistic	df1	df2	Sig.
Initial	.994	3	20	.416
Final	1.227	3	20	.326

Table3 One-way Analysis of Variance (GIC vs GIC-CS) for setting time testing

ANOVA

		Sum of Squares	df	Mean Square	F	Sig.
Initial	Between Groups	210928.125	3	70309.375	74.254	.000
	Within Groups	18937.500	20	946.875		
	Total	229865.625	23			
Final	Between Groups	633375.000	3	211125.000	259.447	.000
	Within Groups	16275.000	20	813.750		
	Total	649650.000	23			

Table 4 Multiple comparison with Scheffe between GIC& GIC-CS for setting time testing

Multiple Comparisons

Scheffe

Dependent Variable	(I) Group	(J) Group	Mean Difference (I-J)	Std. Error	Sig.	95% Confidence Interval Lower Bound	95% Confidence Interval Upper Bound
Initial	GIC	10%CS	-87.50000*	17.76584	.001	-141.6645	-33.3355
		30%CS	-130.00000*	17.76584	.000	-184.1645	-75.8355
		50%CS	-260.00000*	17.76584	.000	-314.1645	-205.8355
	10%CS	GIC	87.50000*	17.76584	.001	33.3355	141.6645
		30%CS	-42.50000	17.76584	.161	-96.6645	11.6645
		50%CS	-172.50000*	17.76584	.000	-226.6645	-118.3355
	30%CS	GIC	130.00000*	17.76584	.000	75.8355	184.1645
		10%CS	42.50000	17.76584	.161	-11.6645	96.6645
		50%CS	-130.00000*	17.76584	.000	-184.1645	-75.8355
	50%CS	GIC	260.00000*	17.76584	.000	205.8355	314.1645
		10%CS	172.50000*	17.76584	.000	118.3355	226.6645
		30%CS	130.00000*	17.76584	.000	75.8355	184.1645
Final	GIC	10%CS	-115.00000*	16.46967	.000	-165.2127	-64.7873
		30%CS	-172.50000*	16.46967	.000	-222.7127	-122.2873
		50%CS	-442.50000*	16.46967	.000	-492.7127	-392.2873
	10%CS	GIC	115.00000*	16.46967	.000	64.7873	165.2127
		30%CS	-57.50000*	16.46967	.021	-107.7127	-7.2873

	50%CS	-327.50000*	16.46967	.000	-377.7127	-277.2873
30%CS	GIC	172.50000*	16.46967	.000	122.2873	222.7127
	10%CS	57.50000*	16.46967	.021	7.2873	107.7127
	50%CS	-270.00000*	16.46967	.000	-320.2127	-219.7873
50%CS	GIC	442.50000*	16.46967	.000	392.2873	492.7127
	10%CS	327.50000*	16.46967	.000	277.2873	377.7127
	30%CS	270.00000*	16.46967	.000	219.7873	320.2127

*. The mean difference is significant at the 0.05 level.

Table 5 Homogeneous subsets for GIC & GIC-CS for setting time testing

Initial

Scheffe[a]

Group	N	Subset for alpha = 0.05		
		1	2	3
GIC	6	265.0000		
10%CS	6		352.5000	
30%CS	6		395.0000	
50%CS	6			525.0000
Sig.		1.000	.161	1.000

Means for groups in homogeneous subsets are displayed.

a. Uses Harmonic Mean Sample Size = 6.000.

Table 6 Independent t-test between GIC-CS(CS10) & MTA for setting time testing

Group Statistics

	Group	N	Mean	Std. Deviation	Std. Error Mean
Initial	10%CS	6	352.5000	32.51923	13.27592
	MTA	6	3840.0000	136.82105	55.85696
Final	10%CS	6	445.0000	33.76389	13.78405
	MTA	6	7290.0000	159.87495	65.26868

Independent Samples Test

		Levene's Test for Equality of Variances		t-test for Equality of Means	
		F	Sig.	t	df
Initial	Equal variances assumed	4.591	.058	-60.744	10
	Equal variances not assumed			-60.744	5.563
Final	Equal variances assumed	11.509	.007	-102.611	10
	Equal variances not assumed			-102.611	5.445

Independent Samples Test

		t-test for Equality of Means		
		Sig. (2-tailed)	Mean Difference	Std. Error Difference
Initial	Equal variances assumed	.000	-3487.50000	57.41298
	Equal variances not assumed	.000	-3487.50000	57.41298
Final	Equal variances assumed	.000	-6845.00000	66.70832
	Equal variances not assumed	.000	-6845.00000	66.70832

Independent Samples Test

		t-test for Equality of Means	
		95% Confidence Interval of the Difference	
		Lower	Upper
Initial	Equal variances assumed	-3615.42409	-3359.57591
	Equal variances not assumed	-3630.70272	-3344.29728
Final	Equal variances assumed	-6993.63540	-6696.36460
	Equal variances not assumed	-7012.34748	-6677.65252

Table 7 Independent t-test between GIC-CS(CS30) & MTA for setting time testing

Group Statistics

	Group	N	Mean	Std. Deviation	Std. Error Mean
Initial	30%CS	6	395.0000	30.98387	12.64911
	MTA	6	3840.0000	136.82105	55.85696
Final	30%CS	6	502.5000	20.67607	8.44097
	MTA	6	7290.0000	159.87495	65.26868

Independent Samples Test

		Levene's Test for Equality of Variances		t-test for Equality of Means	
		F	Sig.	t	df
Initial	Equal variances assumed	5.173	.046	-60.152	10
	Equal variances not assumed			-60.152	5.511
Final	Equal variances assumed	14.593	.003	-103.134	10
	Equal variances not assumed			-103.134	5.167

Independent Samples Test

		t-test for Equality of Means		
		Sig. (2-tailed)	Mean Difference	Std. Error Difference
Initial	Equal variances assumed	.000	-3445.00000	57.27128
	Equal variances not assumed	.000	-3445.00000	57.27128
Final	Equal variances assumed	.000	-6787.50000	65.81223
	Equal variances not assumed	.000	-6787.50000	65.81223

Independent Samples Test

		t-test for Equality of Means 95% Confidence Interval of the Difference	
		Lower	Upper
Initial	Equal variances assumed	-3572.60837	-3317.39163
	Equal variances not assumed	-3588.20458	-3301.79542
Final	Equal variances assumed	-6934.13879	-6640.86121
	Equal variances not assumed	-6955.04234	-6619.95766

Table 8 Independent t-test between GIC-CS(CS50) & MTA for setting time testing

Group Statistics

	Group	N	Mean	Std. Deviation	Std. Error Mean
Initial	50%CS	6	525.0000	40.24922	16.43168
	MTA	6	3840.0000	136.82105	55.85696
Final	50%CS	6	772.5000	35.17812	14.36141
	MTA	6	7290.0000	159.87495	65.26868

Independent Samples Test

		Levene's Test for Equality of Variances		t-test for Equality of Means	
		F	Sig.	t	df
Initial	Equal variances assumed	4.447	.061	-56.936	10
	Equal variances not assumed			-56.936	5.859
Final	Equal variances assumed	11.237	.007	-97.524	10
	Equal variances not assumed			-97.524	5.483

Independent Samples Test

		t-test for Equality of Means		
		Sig. (2-tailed)	Mean Difference	Std. Error Difference
Initial	Equal variances assumed	.000	-3315.00000	58.22371
	Equal variances not assumed	.000	-3315.00000	58.22371
Final	Equal variances assumed	.000	-6517.50000	66.83001
	Equal variances not assumed	.000	-6517.50000	66.83001

Independent Samples Test

		t-test for Equality of Means	
		95% Confidence Interval of the Difference	
		Lower	Upper
Initial	Equal variances assumed	-3444.73050	-3185.26950
	Equal variances not assumed	-3458.30381	-3171.69619
Final	Equal variances assumed	-6666.40654	-6368.59346
	Equal variances not assumed	-6684.83835	-6350.16165

2. Test of pH

Table 9 Descriptive analysis for test of pH

Descriptives

pH

	N	Mean	Std. Deviation	Std. Error	95% Confidence Interval for Mean	
					Lower Bound	Upper Bound
GIC	33	4.1061	.04373	.00761	4.0906	4.1216
10%CS	33	4.3645	.13461	.02343	4.3168	4.4123
30%CS	33	5.7436	.05516	.00960	5.7241	5.7632
50%CS	33	6.4467	.13635	.02374	6.3983	6.4950
MTA	33	12.1939	.08518	.01483	12.1637	12.2241
Total	165	6.5710	2.95206	.22982	6.1172	7.0248

Descriptives

pH

	Minimum	Maximum
GIC	4.02	4.20
10%CS	4.15	4.56
30%CS	5.65	5.81
50%CS	6.22	6.80
MTA	12.00	12.50
Total	4.02	12.50

Table 10 One-way Analysis of Variance for test of pH

ANOVA

pH

	Sum of Squares	df	Mean Square	F	Sig.
Between Groups	1427.640	4	356.910	36477.748	.000
Within Groups	1.565	160	.010		
Total	1429.205	164			

*. The mean difference is significant at the 0.05 level.

Table11 Multiple comparisons between groups for test of pH

pH
Scheffe

(I) group	(J) group	Mean Difference (I-J)	Std. Error	Sig.	95% Confidence Interval Lower Bound	95% Confidence Interval Upper Bound
GIC	10%CS	-.25848*	.02435	.000	-.3344	-.1826
	30%CS	-1.63758*	.02435	.000	-1.7135	-1.5617
	50%CS	-2.34061*	.02435	.000	-2.4165	-2.2647
	MTA	-8.08788*	.02435	.000	-8.1638	-8.0120
10%CS	GIC	.25848*	.02435	.000	.1826	.3344
	30%CS	-1.37909*	.02435	.000	-1.4550	-1.3032
	50%CS	-2.08212*	.02435	.000	-2.1580	-2.0062
	MTA	-7.82939*	.02435	.000	-7.9053	-7.7535
30%CS	GIC	1.63758*	.02435	.000	1.5617	1.7135
	10%CS	1.37909*	.02435	.000	1.3032	1.4550
	50%CS	-.70303*	.02435	.000	-.7789	-.6271
	MTA	-6.45030*	.02435	.000	-6.5262	-6.3744
50%CS	GIC	2.34061*	.02435	.000	2.2647	2.4165
	10%CS	2.08212*	.02435	.000	2.0062	2.1580
	30%CS	.70303*	.02435	.000	.6271	.7789
	MTA	-5.74727*	.02435	.000	-5.8232	-5.6714
MTA	GIC	8.08788*	.02435	.000	8.0120	8.1638
	10%CS	7.82939*	.02435	.000	7.7535	7.9053
	30%CS	6.45030*	.02435	.000	6.3744	6.5262
	50%CS	5.74727*	.02435	.000	5.6714	5.8232

Table 12 Homogeneous subset for test of pH

Homogeneous Subsets

pH

Scheffe[a]

group	N	Subset for alpha = 0.05				
		1	2	3	4	5
GIC	33	4.1061				
10%CS	33		4.3645			
30%CS	33			5.7436		
50%CS	33				6.4467	
MTA	33					12.1939
Sig.		1.000	1.000	1.000	1.000	1.000

Means for groups in homogeneous subsets are displayed.

a. Uses Harmonic Mean Sample Size = 33.000.

3.Compressive strength

Table 13 Descriptives analysis for compressive strength

strength

	N	Mean	Std. Deviation	Std. Error	95% Confidence Interval for Mean		Minimum	Maximum
					Lower Bound	Upper Bound		
gic1	6	66.7167	9.79641	3.99937	56.4360	76.9974	59.50	85.60
gic3	6	84.6667	2.99444	1.22247	81.5242	87.8091	81.00	87.50
gic7	6	103.8500	7.13155	2.91144	96.3659	111.3341	96.80	110.50
gic21	6	126.5000	2.07364	.84656	124.3238	128.6762	125.00	130.00
gic28	6	128.5000	5.08920	2.07766	123.1592	133.8408	125.00	138.00
10cs1	6	61.4500	4.65865	1.90189	56.5610	66.3390	58.90	70.50
10cs3	6	72.9500	6.08104	2.48257	66.5683	79.3317	69.00	80.90
10cs7	6	101.3333	1.94388	.79359	99.2934	103.3733	98.70	103.50
10cs21	6	108.1000	1.75613	.71694	106.2571	109.9429	105.50	110.80
10cs28	6	130.4167	2.45798	1.00347	127.8372	132.9962	128.00	135.00
30cs1	6	77.7333	2.60896	1.06510	74.9954	80.4713	76.00	83.00
30cs3	6	94.4167	8.61056	3.51524	85.3804	103.4529	88.50	106.00
30cs7	6	102.4333	8.84481	3.61088	93.1513	111.7154	95.00	114.00
30cs21	6	103.5833	9.47321	3.86742	93.6418	113.5249	95.50	120.00

30cs28	6	114.8333	7.57408	3.09210	106.8848	122.7818	107.00	123.00
50cs1	6	86.6667	8.61085	3.51536	77.6301	95.7032	77.10	95.50
50cs3	6	75.2500	4.58290	1.87096	70.4405	80.0595	66.20	78.50
50cs7	6	96.6833	6.25921	2.55531	90.1147	103.2520	90.00	105.00
50cs21	6	80.3000	7.12432	2.90849	72.8235	87.7765	70.50	86.50
50cs28	6	80.9667	4.69667	1.91741	76.0378	85.8955	74.20	86.50
mta1	6	30.9833	4.99737	2.04017	25.7389	36.2277	24.40	38.80
mta3	6	66.4333	10.33241	4.21819	55.5901	77.2765	55.50	79.50
mta7	6	66.4500	2.45662	1.00291	63.8719	69.0281	63.30	68.80
mta21	6	114.8333	8.13429	3.32081	106.2969	123.3697	104.00	129.00
mta28	6	122.3333	7.23095	2.95202	114.7449	129.9218	113.00	132.30
Total	150	91.9353	24.87380	2.03094	87.9222	95.9485	24.40	138.00

Table 14 One-way Analysis of Variance for compressive strength

ANOVA

strength

	Sum of Squares	df	Mean Square	F	Sig.
Between Groups	87051.024	24	3627.126	88.274	.000
Within Groups	5136.158	125	41.089		
Total	92187.183	149			

Table 15 Multiple comparison(Post-Hoc test:Scheffe) for compressive strength

Multiple Comparisons

(I) group.time	(J) group.time	Mean Difference (I-J)	Std. Error	Sig.	95% Confidence Interval Lower Bound	95% Confidence Interval Upper Bound
gic1	gic3	-17.95000	3.70087	.497	-40.9177	5.0177
	gic7	-37.13333*	3.70087	.000	-60.1010	-14.1656
	gic21	-59.78333*	3.70087	.000	-82.7510	-36.8156
	gic28	-61.78333*	3.70087	.000	-84.7510	-38.8156
	10cs1	5.26667	3.70087	1.000	-17.7010	28.2344

	10cs3	-6.23333	3.70087	1.000	-29.2010	16.7344
	10cs7	-34.61667*	3.70087	.000	-57.5844	-11.6490
	10cs21	-41.38333*	3.70087	.000	-64.3510	-18.4156
	10cs28	-63.70000*	3.70087	.000	-86.6677	-40.7323
	30cs1	-11.01667	3.70087	.997	-33.9844	11.9510
	30cs3	-27.70000*	3.70087	.001	-50.6677	-4.7323
	30cs7	-35.71667*	3.70087	.000	-58.6844	-12.7490
	30cs21	-36.86667*	3.70087	.000	-59.8344	-13.8990
	30cs28	-48.11667*	3.70087	.000	-71.0844	-25.1490
	50cs1	-19.95000	3.70087	.246	-42.9177	3.0177
	50cs3	-8.53333	3.70087	1.000	-31.5010	14.4344
	50cs7	-29.96667*	3.70087	.000	-52.9344	-6.9990
	50cs21	-13.58333	3.70087	.949	-36.5510	9.3844
	50cs28	-14.25000	3.70087	.914	-37.2177	8.7177
	mta1	35.73333*	3.70087	.000	12.7656	58.7010
	mta3	.28333	3.70087	1.000	-22.6844	23.2510
	mta7	.26667	3.70087	1.000	-22.7010	23.2344
	mta21	-48.11667*	3.70087	.000	-71.0844	-25.1490
	mta28	-55.61667*	3.70087	.000	-78.5844	-32.6490
gic3	gic1	17.95000	3.70087	.497	-5.0177	40.9177
	gic7	-19.18333	3.70087	.333	-42.1510	3.7844
	gic21	-41.83333*	3.70087	.000	-64.8010	-18.8656
	gic28	-43.83333*	3.70087	.000	-66.8010	-20.8656
	10cs1	23.21667*	3.70087	.043	.2490	46.1844
	10cs3	11.71667	3.70087	.992	-11.2510	34.6844
	10cs7	-16.66667	3.70087	.674	-39.6344	6.3010
	10cs21	-23.43333*	3.70087	.037	-46.4010	-.4656
	10cs28	-45.75000*	3.70087	.000	-68.7177	-22.7823
	30cs1	6.93333	3.70087	1.000	-16.0344	29.9010
	30cs3	-9.75000	3.70087	1.000	-32.7177	13.2177
	30cs7	-17.76667	3.70087	.522	-40.7344	5.2010
	30cs21	-18.91667	3.70087	.366	-41.8844	4.0510
	30cs28	-30.16667*	3.70087	.000	-53.1344	-7.1990
	50cs1	-2.00000	3.70087	1.000	-24.9677	20.9677
	50cs3	9.41667	3.70087	1.000	-13.5510	32.3844
	50cs7	-12.01667	3.70087	.989	-34.9844	10.9510

	50cs21	4.36667	3.70087	1.000	-18.6010	27.3344
	50cs28	3.70000	3.70087	1.000	-19.2677	26.6677
	mta1	53.68333*	3.70087	.000	30.7156	76.6510
	mta3	18.23333	3.70087	.457	-4.7344	41.2010
	mta7	18.21667	3.70087	.460	-4.7510	41.1844
	mta21	-30.16667*	3.70087	.000	-53.1344	-7.1990
	mta28	-37.66667*	3.70087	.000	-60.6344	-14.6990
gic7	gic1	37.13333*	3.70087	.000	14.1656	60.1010
	gic3	19.18333	3.70087	.333	-3.7844	42.1510
	gic21	-22.65000	3.70087	.061	-45.6177	.3177
	gic28	-24.65000*	3.70087	.016	-47.6177	-1.6823
	10cs1	42.40000*	3.70087	.000	19.4323	65.3677
	10cs3	30.90000*	3.70087	.000	7.9323	53.8677
	10cs7	2.51667	3.70087	1.000	-20.4510	25.4844
	10cs21	-4.25000	3.70087	1.000	-27.2177	18.7177
	10cs28	-26.56667*	3.70087	.004	-49.5344	-3.5990
	30cs1	26.11667*	3.70087	.005	3.1490	49.0844
	30cs3	9.43333	3.70087	1.000	-13.5344	32.4010
	30cs7	1.41667	3.70087	1.000	-21.5510	24.3844
	30cs21	.26667	3.70087	1.000	-22.7010	23.2344
	30cs28	-10.98333	3.70087	.997	-33.9510	11.9844
	50cs1	17.18333	3.70087	.604	-5.7844	40.1510
	50cs3	28.60000*	3.70087	.001	5.6323	51.5677
	50cs7	7.16667	3.70087	1.000	-15.8010	30.1344
	50cs21	23.55000*	3.70087	.034	.5823	46.5177
	50cs28	22.88333	3.70087	.053	-.0844	45.8510
	mta1	72.86667*	3.70087	.000	49.8990	95.8344
	mta3	37.41667*	3.70087	.000	14.4490	60.3844
	mta7	37.40000*	3.70087	.000	14.4323	60.3677
	mta21	-10.98333	3.70087	.997	-33.9510	11.9844
	mta28	-18.48333	3.70087	.423	-41.4510	4.4844
gic21	gic1	59.78333*	3.70087	.000	36.8156	82.7510
	gic3	41.83333*	3.70087	.000	18.8656	64.8010
	gic7	22.65000	3.70087	.061	-.3177	45.6177
	gic28	-2.00000	3.70087	1.000	-24.9677	20.9677
	10cs1	65.05000*	3.70087	.000	42.0823	88.0177
	10cs3	53.55000*	3.70087	.000	30.5823	76.5177

	10cs7	25.16667*	3.70087	.011	2.1990	48.1344
	10cs21	18.40000	3.70087	.434	-4.5677	41.3677
	10cs28	-3.91667	3.70087	1.000	-26.8844	19.0510
	30cs1	48.76667*	3.70087	.000	25.7990	71.7344
	30cs3	32.08333*	3.70087	.000	9.1156	55.0510
	30cs7	24.06667*	3.70087	.024	1.0990	47.0344
	30cs21	22.91667	3.70087	.052	-.0510	45.8844
	30cs28	11.66667	3.70087	.993	-11.3010	34.6344
	50cs1	39.83333*	3.70087	.000	16.8656	62.8010
	50cs3	51.25000*	3.70087	.000	28.2823	74.2177
	50cs7	29.81667*	3.70087	.000	6.8490	52.7844
	50cs21	46.20000*	3.70087	.000	23.2323	69.1677
	50cs28	45.53333*	3.70087	.000	22.5656	68.5010
	mta1	95.51667*	3.70087	.000	72.5490	118.4844
	mta3	60.06667*	3.70087	.000	37.0990	83.0344
	mta7	60.05000*	3.70087	.000	37.0823	83.0177
	mta21	11.66667	3.70087	.993	-11.3010	34.6344
	mta28	4.16667	3.70087	1.000	-18.8010	27.1344
gic28	gic1	61.78333*	3.70087	.000	38.8156	84.7510
	gic3	43.83333*	3.70087	.000	20.8656	66.8010
	gic7	24.65000*	3.70087	.016	1.6823	47.6177
	gic21	2.00000	3.70087	1.000	-20.9677	24.9677
	10cs1	67.05000*	3.70087	.000	44.0823	90.0177
	10cs3	55.55000*	3.70087	.000	32.5823	78.5177
	10cs7	27.16667*	3.70087	.002	4.1990	50.1344
	10cs21	20.40000	3.70087	.202	-2.5677	43.3677
	10cs28	-1.91667	3.70087	1.000	-24.8844	21.0510
	30cs1	50.76667*	3.70087	.000	27.7990	73.7344
	30cs3	34.08333*	3.70087	.000	11.1156	57.0510
	30cs7	26.06667*	3.70087	.005	3.0990	49.0344
	30cs21	24.91667*	3.70087	.013	1.9490	47.8844
	30cs28	13.66667	3.70087	.945	-9.3010	36.6344
	50cs1	41.83333*	3.70087	.000	18.8656	64.8010
	50cs3	53.25000*	3.70087	.000	30.2823	76.2177
	50cs7	31.81667*	3.70087	.000	8.8490	54.7844
	50cs21	48.20000*	3.70087	.000	25.2323	71.1677
	50cs28	47.53333*	3.70087	.000	24.5656	70.5010

	mta1	97.51667*	3.70087	.000	74.5490	120.4844
	mta3	62.06667*	3.70087	.000	39.0990	85.0344
	mta7	62.05000*	3.70087	.000	39.0823	85.0177
	mta21	13.66667	3.70087	.945	-9.3010	36.6344
	mta28	6.16667	3.70087	1.000	-16.8010	29.1344
10cs1	gic1	-5.26667	3.70087	1.000	-28.2344	17.7010
	gic3	-23.21667*	3.70087	.043	-46.1844	-.2490
	gic7	-42.40000*	3.70087	.000	-65.3677	-19.4323
	gic21	-65.05000*	3.70087	.000	-88.0177	-42.0823
	gic28	-67.05000*	3.70087	.000	-90.0177	-44.0823
	10cs3	-11.50000	3.70087	.994	-34.4677	11.4677
	10cs7	-39.88333*	3.70087	.000	-62.8510	-16.9156
	10cs21	-46.65000*	3.70087	.000	-69.6177	-23.6823
	10cs28	-68.96667*	3.70087	.000	-91.9344	-45.9990
	30cs1	-16.28333	3.70087	.723	-39.2510	6.6844
	30cs3	-32.96667*	3.70087	.000	-55.9344	-9.9990
	30cs7	-40.98333*	3.70087	.000	-63.9510	-18.0156
	30cs21	-42.13333*	3.70087	.000	-65.1010	-19.1656
	30cs28	-53.38333*	3.70087	.000	-76.3510	-30.4156
	50cs1	-25.21667*	3.70087	.010	-48.1844	-2.2490
	50cs3	-13.80000	3.70087	.939	-36.7677	9.1677
	50cs7	-35.23333*	3.70087	.000	-58.2010	-12.2656
	50cs21	-18.85000	3.70087	.375	-41.8177	4.1177
	50cs28	-19.51667	3.70087	.293	-42.4844	3.4510
	mta1	30.46667*	3.70087	.000	7.4990	53.4344
	mta3	-4.98333	3.70087	1.000	-27.9510	17.9844
	mta7	-5.00000	3.70087	1.000	-27.9677	17.9677
	mta21	-53.38333*	3.70087	.000	-76.3510	-30.4156
	mta28	-60.88333*	3.70087	.000	-83.8510	-37.9156
10cs3	gic1	6.23333	3.70087	1.000	-16.7344	29.2010
	gic3	-11.71667	3.70087	.992	-34.6844	11.2510
	gic7	-30.90000*	3.70087	.000	-53.8677	-7.9323
	gic21	-53.55000*	3.70087	.000	-76.5177	-30.5823
	gic28	-55.55000*	3.70087	.000	-78.5177	-32.5823
	10cs1	11.50000	3.70087	.994	-11.4677	34.4677
	10cs7	-28.38333*	3.70087	.001	-51.3510	-5.4156
	10cs21	-35.15000*	3.70087	.000	-58.1177	-12.1823

	10cs28	-57.46667*	3.70087	.000	-80.4344	-34.4990
	30cs1	-4.78333	3.70087	1.000	-27.7510	18.1844
	30cs3	-21.46667	3.70087	.119	-44.4344	1.5010
	30cs7	-29.48333*	3.70087	.000	-52.4510	-6.5156
	30cs21	-30.63333*	3.70087	.000	-53.6010	-7.6656
	30cs28	-41.88333*	3.70087	.000	-64.8510	-18.9156
	50cs1	-13.71667	3.70087	.943	-36.6844	9.2510
	50cs3	-2.30000	3.70087	1.000	-25.2677	20.6677
	50cs7	-23.73333*	3.70087	.030	-46.7010	-.7656
	50cs21	-7.35000	3.70087	1.000	-30.3177	15.6177
	50cs28	-8.01667	3.70087	1.000	-30.9844	14.9510
	mta1	41.96667*	3.70087	.000	18.9990	64.9344
	mta3	6.51667	3.70087	1.000	-16.4510	29.4844
	mta7	6.50000	3.70087	1.000	-16.4677	29.4677
	mta21	-41.88333*	3.70087	.000	-64.8510	-18.9156
	mta28	-49.38333*	3.70087	.000	-72.3510	-26.4156
10cs7	gic1	34.61667*	3.70087	.000	11.6490	57.5844
	gic3	16.66667	3.70087	.674	-6.3010	39.6344
	gic7	-2.51667	3.70087	1.000	-25.4844	20.4510
	gic21	-25.16667*	3.70087	.011	-48.1344	-2.1990
	gic28	-27.16667*	3.70087	.002	-50.1344	-4.1990
	10cs1	39.88333*	3.70087	.000	16.9156	62.8510
	10cs3	28.38333*	3.70087	.001	5.4156	51.3510
	10cs21	-6.76667	3.70087	1.000	-29.7344	16.2010
	10cs28	-29.08333*	3.70087	.000	-52.0510	-6.1156
	30cs1	23.60000*	3.70087	.033	.6323	46.5677
	30cs3	6.91667	3.70087	1.000	-16.0510	29.8844
	30cs7	-1.10000	3.70087	1.000	-24.0677	21.8677
	30cs21	-2.25000	3.70087	1.000	-25.2177	20.7177
	30cs28	-13.50000	3.70087	.953	-36.4677	9.4677
	50cs1	14.66667	3.70087	.886	-8.3010	37.6344
	50cs3	26.08333*	3.70087	.005	3.1156	49.0510
	50cs7	4.65000	3.70087	1.000	-18.3177	27.6177
	50cs21	21.03333	3.70087	.149	-1.9344	44.0010
	50cs28	20.36667	3.70087	.205	-2.6010	43.3344
	mta1	70.35000*	3.70087	.000	47.3823	93.3177
	mta3	34.90000*	3.70087	.000	11.9323	57.8677

	mta7	34.88333*	3.70087	.000	11.9156	57.8510
	mta21	-13.50000	3.70087	.953	-36.4677	9.4677
	mta28	-21.00000	3.70087	.151	-43.9677	1.9677
10cs21	gic1	41.38333*	3.70087	.000	18.4156	64.3510
	gic3	23.43333*	3.70087	.037	.4656	46.4010
	gic7	4.25000	3.70087	1.000	-18.7177	27.2177
	gic21	-18.40000	3.70087	.434	-41.3677	4.5677
	gic28	-20.40000	3.70087	.202	-43.3677	2.5677
	10cs1	46.65000*	3.70087	.000	23.6823	69.6177
	10cs3	35.15000*	3.70087	.000	12.1823	58.1177
	10cs7	6.76667	3.70087	1.000	-16.2010	29.7344
	10cs28	-22.31667	3.70087	.074	-45.2844	.6510
	30cs1	30.36667*	3.70087	.000	7.3990	53.3344
	30cs3	13.68333	3.70087	.945	-9.2844	36.6510
	30cs7	5.66667	3.70087	1.000	-17.3010	28.6344
	30cs21	4.51667	3.70087	1.000	-18.4510	27.4844
	30cs28	-6.73333	3.70087	1.000	-29.7010	16.2344
	50cs1	21.43333	3.70087	.121	-1.5344	44.4010
	50cs3	32.85000*	3.70087	.000	9.8823	55.8177
	50cs7	11.41667	3.70087	.995	-11.5510	34.3844
	50cs21	27.80000*	3.70087	.001	4.8323	50.7677
	50cs28	27.13333*	3.70087	.002	4.1656	50.1010
	mta1	77.11667*	3.70087	.000	54.1490	100.0844
	mta3	41.66667*	3.70087	.000	18.6990	64.6344
	mta7	41.65000*	3.70087	.000	18.6823	64.6177
	mta21	-6.73333	3.70087	1.000	-29.7010	16.2344
	mta28	-14.23333	3.70087	.915	-37.2010	8.7344
10cs28	gic1	63.70000*	3.70087	.000	40.7323	86.6677
	gic3	45.75000*	3.70087	.000	22.7823	68.7177
	gic7	26.56667*	3.70087	.004	3.5990	49.5344
	gic21	3.91667	3.70087	1.000	-19.0510	26.8844
	gic28	1.91667	3.70087	1.000	-21.0510	24.8844
	10cs1	68.96667*	3.70087	.000	45.9990	91.9344
	10cs3	57.46667*	3.70087	.000	34.4990	80.4344
	10cs7	29.08333*	3.70087	.000	6.1156	52.0510
	10cs21	22.31667	3.70087	.074	-.6510	45.2844
	30cs1	52.68333*	3.70087	.000	29.7156	75.6510

	30cs3	36.00000*	3.70087	.000	13.0323	58.9677
	30cs7	27.98333*	3.70087	.001	5.0156	50.9510
	30cs21	26.83333*	3.70087	.003	3.8656	49.8010
	30cs28	15.58333	3.70087	.803	-7.3844	38.5510
	50cs1	43.75000*	3.70087	.000	20.7823	66.7177
	50cs3	55.16667*	3.70087	.000	32.1990	78.1344
	50cs7	33.73333*	3.70087	.000	10.7656	56.7010
	50cs21	50.11667*	3.70087	.000	27.1490	73.0844
	50cs28	49.45000*	3.70087	.000	26.4823	72.4177
	mta1	99.43333*	3.70087	.000	76.4656	122.4010
	mta3	63.98333*	3.70087	.000	41.0156	86.9510
	mta7	63.96667*	3.70087	.000	40.9990	86.9344
	mta21	15.58333	3.70087	.803	-7.3844	38.5510
	mta28	8.08333	3.70087	1.000	-14.8844	31.0510
30cs1	gic1	11.01667	3.70087	.997	-11.9510	33.9844
	gic3	-6.93333	3.70087	1.000	-29.9010	16.0344
	gic7	-26.11667*	3.70087	.005	-49.0844	-3.1490
	gic21	-48.76667*	3.70087	.000	-71.7344	-25.7990
	gic28	-50.76667*	3.70087	.000	-73.7344	-27.7990
	10cs1	16.28333	3.70087	.723	-6.6844	39.2510
	10cs3	4.78333	3.70087	1.000	-18.1844	27.7510
	10cs7	-23.60000*	3.70087	.033	-46.5677	-.6323
	10cs21	-30.36667*	3.70087	.000	-53.3344	-7.3990
	10cs28	-52.68333*	3.70087	.000	-75.6510	-29.7156
	30cs3	-16.68333	3.70087	.672	-39.6510	6.2844
	30cs7	-24.70000*	3.70087	.015	-47.6677	-1.7323
	30cs21	-25.85000*	3.70087	.006	-48.8177	-2.8823
	30cs28	-37.10000*	3.70087	.000	-60.0677	-14.1323
	50cs1	-8.93333	3.70087	1.000	-31.9010	14.0344
	50cs3	2.48333	3.70087	1.000	-20.4844	25.4510
	50cs7	-18.95000	3.70087	.362	-41.9177	4.0177
	50cs21	-2.56667	3.70087	1.000	-25.5344	20.4010
	50cs28	-3.23333	3.70087	1.000	-26.2010	19.7344
	mta1	46.75000*	3.70087	.000	23.7823	69.7177
	mta3	11.30000	3.70087	.995	-11.6677	34.2677
	mta7	11.28333	3.70087	.996	-11.6844	34.2510
	mta21	-37.10000*	3.70087	.000	-60.0677	-14.1323

	mta28	-44.60000*	3.70087	.000	-67.5677	-21.6323
30cs3	gic1	27.70000*	3.70087	.001	4.7323	50.6677
	gic3	9.75000	3.70087	1.000	-13.2177	32.7177
	gic7	-9.43333	3.70087	1.000	-32.4010	13.5344
	gic21	-32.08333*	3.70087	.000	-55.0510	-9.1156
	gic28	-34.08333*	3.70087	.000	-57.0510	-11.1156
	10cs1	32.96667*	3.70087	.000	9.9990	55.9344
	10cs3	21.46667	3.70087	.119	-1.5010	44.4344
	10cs7	-6.91667	3.70087	1.000	-29.8844	16.0510
	10cs21	-13.68333	3.70087	.945	-36.6510	9.2844
	10cs28	-36.00000*	3.70087	.000	-58.9677	-13.0323
	30cs1	16.68333	3.70087	.672	-6.2844	39.6510
	30cs7	-8.01667	3.70087	1.000	-30.9844	14.9510
	30cs21	-9.16667	3.70087	1.000	-32.1344	13.8010
	30cs28	-20.41667	3.70087	.200	-43.3844	2.5510
	50cs1	7.75000	3.70087	1.000	-15.2177	30.7177
	50cs3	19.16667	3.70087	.335	-3.8010	42.1344
	50cs7	-2.26667	3.70087	1.000	-25.2344	20.7010
	50cs21	14.11667	3.70087	.922	-8.8510	37.0844
	50cs28	13.45000	3.70087	.954	-9.5177	36.4177
	mta1	63.43333*	3.70087	.000	40.4656	86.4010
	mta3	27.98333*	3.70087	.001	5.0156	50.9510
	mta7	27.96667*	3.70087	.001	4.9990	50.9344
	mta21	-20.41667	3.70087	.200	-43.3844	2.5510
	mta28	-27.91667*	3.70087	.001	-50.8844	-4.9490
30cs7	gic1	35.71667*	3.70087	.000	12.7490	58.6844
	gic3	17.76667	3.70087	.522	-5.2010	40.7344
	gic7	-1.41667	3.70087	1.000	-24.3844	21.5510
	gic21	-24.06667*	3.70087	.024	-47.0344	-1.0990
	gic28	-26.06667*	3.70087	.005	-49.0344	-3.0990
	10cs1	40.98333*	3.70087	.000	18.0156	63.9510
	10cs3	29.48333*	3.70087	.000	6.5156	52.4510
	10cs7	1.10000	3.70087	1.000	-21.8677	24.0677
	10cs21	-5.66667	3.70087	1.000	-28.6344	17.3010
	10cs28	-27.98333*	3.70087	.001	-50.9510	-5.0156
	30cs1	24.70000*	3.70087	.015	1.7323	47.6677
	30cs3	8.01667	3.70087	1.000	-14.9510	30.9844

	30cs21	-1.15000	3.70087	1.000	-24.1177	21.8177
	30cs28	-12.40000	3.70087	.983	-35.3677	10.5677
	50cs1	15.76667	3.70087	.783	-7.2010	38.7344
	50cs3	27.18333*	3.70087	.002	4.2156	50.1510
	50cs7	5.75000	3.70087	1.000	-17.2177	28.7177
	50cs21	22.13333	3.70087	.083	-.8344	45.1010
	50cs28	21.46667	3.70087	.119	-1.5010	44.4344
	mta1	71.45000*	3.70087	.000	48.4823	94.4177
	mta3	36.00000*	3.70087	.000	13.0323	58.9677
	mta7	35.98333*	3.70087	.000	13.0156	58.9510
	mta21	-12.40000	3.70087	.983	-35.3677	10.5677
	mta28	-19.90000	3.70087	.251	-42.8677	3.0677
30cs21	gic1	36.86667*	3.70087	.000	13.8990	59.8344
	gic3	18.91667	3.70087	.366	-4.0510	41.8844
	gic7	-.26667	3.70087	1.000	-23.2344	22.7010
	gic21	-22.91667	3.70087	.052	-45.8844	.0510
	gic28	-24.91667*	3.70087	.013	-47.8844	-1.9490
	10cs1	42.13333*	3.70087	.000	19.1656	65.1010
	10cs3	30.63333*	3.70087	.000	7.6656	53.6010
	10cs7	2.25000	3.70087	1.000	-20.7177	25.2177
	10cs21	-4.51667	3.70087	1.000	-27.4844	18.4510
	10cs28	-26.83333*	3.70087	.003	-49.8010	-3.8656
	30cs1	25.85000*	3.70087	.006	2.8823	48.8177
	30cs3	9.16667	3.70087	1.000	-13.8010	32.1344
	30cs7	1.15000	3.70087	1.000	-21.8177	24.1177
	30cs28	-11.25000	3.70087	.996	-34.2177	11.7177
	50cs1	16.91667	3.70087	.640	-6.0510	39.8844
	50cs3	28.33333*	3.70087	.001	5.3656	51.3010
	50cs7	6.90000	3.70087	1.000	-16.0677	29.8677
	50cs21	23.28333*	3.70087	.041	.3156	46.2510
	50cs28	22.61667	3.70087	.062	-.3510	45.5844
	mta1	72.60000*	3.70087	.000	49.6323	95.5677
	mta3	37.15000*	3.70087	.000	14.1823	60.1177
	mta7	37.13333*	3.70087	.000	14.1656	60.1010
	mta21	-11.25000	3.70087	.996	-34.2177	11.7177
	mta28	-18.75000	3.70087	.388	-41.7177	4.2177
30cs28	gic1	48.11667*	3.70087	.000	25.1490	71.0844

	gic3	30.16667*	3.70087	.000	7.1990	53.1344
	gic7	10.98333	3.70087	.997	-11.9844	33.9510
	gic21	-11.66667	3.70087	.993	-34.6344	11.3010
	gic28	-13.66667	3.70087	.945	-36.6344	9.3010
	10cs1	53.38333*	3.70087	.000	30.4156	76.3510
	10cs3	41.88333*	3.70087	.000	18.9156	64.8510
	10cs7	13.50000	3.70087	.953	-9.4677	36.4677
	10cs21	6.73333	3.70087	1.000	-16.2344	29.7010
	10cs28	-15.58333	3.70087	.803	-38.5510	7.3844
	30cs1	37.10000*	3.70087	.000	14.1323	60.0677
	30cs3	20.41667	3.70087	.200	-2.5510	43.3844
	30cs7	12.40000	3.70087	.983	-10.5677	35.3677
	30cs21	11.25000	3.70087	.996	-11.7177	34.2177
	50cs1	28.16667*	3.70087	.001	5.1990	51.1344
	50cs3	39.58333*	3.70087	.000	16.6156	62.5510
	50cs7	18.15000	3.70087	.469	-4.8177	41.1177
	50cs21	34.53333*	3.70087	.000	11.5656	57.5010
	50cs28	33.86667*	3.70087	.000	10.8990	56.8344
	mta1	83.85000*	3.70087	.000	60.8823	106.8177
	mta3	48.40000*	3.70087	.000	25.4323	71.3677
	mta7	48.38333*	3.70087	.000	25.4156	71.3510
	mta21	.00000	3.70087	1.000	-22.9677	22.9677
	mta28	-7.50000	3.70087	1.000	-30.4677	15.4677
50cs1	gic1	19.95000	3.70087	.246	-3.0177	42.9177
	gic3	2.00000	3.70087	1.000	-20.9677	24.9677
	gic7	-17.18333	3.70087	.604	-40.1510	5.7844
	gic21	-39.83333*	3.70087	.000	-62.8010	-16.8656
	gic28	-41.83333*	3.70087	.000	-64.8010	-18.8656
	10cs1	25.21667*	3.70087	.010	2.2490	48.1844
	10cs3	13.71667	3.70087	.943	-9.2510	36.6844
	10cs7	-14.66667	3.70087	.886	-37.6344	8.3010
	10cs21	-21.43333	3.70087	.121	-44.4010	1.5344
	10cs28	-43.75000*	3.70087	.000	-66.7177	-20.7823
	30cs1	8.93333	3.70087	1.000	-14.0344	31.9010
	30cs3	-7.75000	3.70087	1.000	-30.7177	15.2177
	30cs7	-15.76667	3.70087	.783	-38.7344	7.2010
	30cs21	-16.91667	3.70087	.640	-39.8844	6.0510

	30cs28	-28.16667*	3.70087	.001	-51.1344	-5.1990
	50cs3	11.41667	3.70087	.995	-11.5510	34.3844
	50cs7	-10.01667	3.70087	.999	-32.9844	12.9510
	50cs21	6.36667	3.70087	1.000	-16.6010	29.3344
	50cs28	5.70000	3.70087	1.000	-17.2677	28.6677
	mta1	55.68333*	3.70087	.000	32.7156	78.6510
	mta3	20.23333	3.70087	.217	-2.7344	43.2010
	mta7	20.21667	3.70087	.219	-2.7510	43.1844
	mta21	-28.16667*	3.70087	.001	-51.1344	-5.1990
	mta28	-35.66667*	3.70087	.000	-58.6344	-12.6990
50cs3	gic1	8.53333	3.70087	1.000	-14.4344	31.5010
	gic3	-9.41667	3.70087	1.000	-32.3844	13.5510
	gic7	-28.60000*	3.70087	.001	-51.5677	-5.6323
	gic21	-51.25000*	3.70087	.000	-74.2177	-28.2823
	gic28	-53.25000*	3.70087	.000	-76.2177	-30.2823
	10cs1	13.80000	3.70087	.939	-9.1677	36.7677
	10cs3	2.30000	3.70087	1.000	-20.6677	25.2677
	10cs7	-26.08333*	3.70087	.005	-49.0510	-3.1156
	10cs21	-32.85000*	3.70087	.000	-55.8177	-9.8823
	10cs28	-55.16667*	3.70087	.000	-78.1344	-32.1990
	30cs1	-2.48333	3.70087	1.000	-25.4510	20.4844
	30cs3	-19.16667	3.70087	.335	-42.1344	3.8010
	30cs7	-27.18333*	3.70087	.002	-50.1510	-4.2156
	30cs21	-28.33333*	3.70087	.001	-51.3010	-5.3656
	30cs28	-39.58333*	3.70087	.000	-62.5510	-16.6156
	50cs1	-11.41667	3.70087	.995	-34.3844	11.5510
	50cs7	-21.43333	3.70087	.121	-44.4010	1.5344
	50cs21	-5.05000	3.70087	1.000	-28.0177	17.9177
	50cs28	-5.71667	3.70087	1.000	-28.6844	17.2510
	mta1	44.26667*	3.70087	.000	21.2990	67.2344
	mta3	8.81667	3.70087	1.000	-14.1510	31.7844
	mta7	8.80000	3.70087	1.000	-14.1677	31.7677
	mta21	-39.58333*	3.70087	.000	-62.5510	-16.6156
	mta28	-47.08333*	3.70087	.000	-70.0510	-24.1156
50cs7	gic1	29.96667*	3.70087	.000	6.9990	52.9344
	gic3	12.01667	3.70087	.989	-10.9510	34.9844
	gic7	-7.16667	3.70087	1.000	-30.1344	15.8010

	gic21	-29.81667*	3.70087	.000	-52.7844	-6.8490
	gic28	-31.81667*	3.70087	.000	-54.7844	-8.8490
	10cs1	35.23333*	3.70087	.000	12.2656	58.2010
	10cs3	23.73333*	3.70087	.030	.7656	46.7010
	10cs7	-4.65000	3.70087	1.000	-27.6177	18.3177
	10cs21	-11.41667	3.70087	.995	-34.3844	11.5510
	10cs28	-33.73333*	3.70087	.000	-56.7010	-10.7656
	30cs1	18.95000	3.70087	.362	-4.0177	41.9177
	30cs3	2.26667	3.70087	1.000	-20.7010	25.2344
	30cs7	-5.75000	3.70087	1.000	-28.7177	17.2177
	30cs21	-6.90000	3.70087	1.000	-29.8677	16.0677
	30cs28	-18.15000	3.70087	.469	-41.1177	4.8177
	50cs1	10.01667	3.70087	.999	-12.9510	32.9844
	50cs3	21.43333	3.70087	.121	-1.5344	44.4010
	50cs21	16.38333	3.70087	.710	-6.5844	39.3510
	50cs28	15.71667	3.70087	.789	-7.2510	38.6844
	mta1	65.70000*	3.70087	.000	42.7323	88.6677
	mta3	30.25000*	3.70087	.000	7.2823	53.2177
	mta7	30.23333*	3.70087	.000	7.2656	53.2010
	mta21	-18.15000	3.70087	.469	-41.1177	4.8177
	mta28	-25.65000*	3.70087	.007	-48.6177	-2.6823
50cs21	gic1	13.58333	3.70087	.949	-9.3844	36.5510
	gic3	-4.36667	3.70087	1.000	-27.3344	18.6010
	gic7	-23.55000*	3.70087	.034	-46.5177	-.5823
	gic21	-46.20000*	3.70087	.000	-69.1677	-23.2323
	gic28	-48.20000*	3.70087	.000	-71.1677	-25.2323
	10cs1	18.85000	3.70087	.375	-4.1177	41.8177
	10cs3	7.35000	3.70087	1.000	-15.6177	30.3177
	10cs7	-21.03333	3.70087	.149	-44.0010	1.9344
	10cs21	-27.80000*	3.70087	.001	-50.7677	-4.8323
	10cs28	-50.11667*	3.70087	.000	-73.0844	-27.1490
	30cs1	2.56667	3.70087	1.000	-20.4010	25.5344
	30cs3	-14.11667	3.70087	.922	-37.0844	8.8510
	30cs7	-22.13333	3.70087	.083	-45.1010	.8344
	30cs21	-23.28333*	3.70087	.041	-46.2510	-.3156
	30cs28	-34.53333*	3.70087	.000	-57.5010	-11.5656
	50cs1	-6.36667	3.70087	1.000	-29.3344	16.6010

	50cs3	5.05000	3.70087	1.000	-17.9177	28.0177
	50cs7	-16.38333	3.70087	.710	-39.3510	6.5844
	50cs28	-.66667	3.70087	1.000	-23.6344	22.3010
	mta1	49.31667*	3.70087	.000	26.3490	72.2844
	mta3	13.86667	3.70087	.936	-9.1010	36.8344
	mta7	13.85000	3.70087	.937	-9.1177	36.8177
	mta21	-34.53333*	3.70087	.000	-57.5010	-11.5656
	mta28	-42.03333*	3.70087	.000	-65.0010	-19.0656
50cs28	gic1	14.25000	3.70087	.914	-8.7177	37.2177
	gic3	-3.70000	3.70087	1.000	-26.6677	19.2677
	gic7	-22.88333	3.70087	.053	-45.8510	.0844
	gic21	-45.53333*	3.70087	.000	-68.5010	-22.5656
	gic28	-47.53333*	3.70087	.000	-70.5010	-24.5656
	10cs1	19.51667	3.70087	.293	-3.4510	42.4844
	10cs3	8.01667	3.70087	1.000	-14.9510	30.9844
	10cs7	-20.36667	3.70087	.205	-43.3344	2.6010
	10cs21	-27.13333*	3.70087	.002	-50.1010	-4.1656
	10cs28	-49.45000*	3.70087	.000	-72.4177	-26.4823
	30cs1	3.23333	3.70087	1.000	-19.7344	26.2010
	30cs3	-13.45000	3.70087	.954	-36.4177	9.5177
	30cs7	-21.46667	3.70087	.119	-44.4344	1.5010
	30cs21	-22.61667	3.70087	.062	-45.5844	.3510
	30cs28	-33.86667*	3.70087	.000	-56.8344	-10.8990
	50cs1	-5.70000	3.70087	1.000	-28.6677	17.2677
	50cs3	5.71667	3.70087	1.000	-17.2510	28.6844
	50cs7	-15.71667	3.70087	.789	-38.6844	7.2510
	50cs21	.66667	3.70087	1.000	-22.3010	23.6344
	mta1	49.98333*	3.70087	.000	27.0156	72.9510
	mta3	14.53333	3.70087	.896	-8.4344	37.5010
	mta7	14.51667	3.70087	.897	-8.4510	37.4844
	mta21	-33.86667*	3.70087	.000	-56.8344	-10.8990
	mta28	-41.36667*	3.70087	.000	-64.3344	-18.3990
mta1	gic1	-35.73333*	3.70087	.000	-58.7010	-12.7656
	gic3	-53.68333*	3.70087	.000	-76.6510	-30.7156
	gic7	-72.86667*	3.70087	.000	-95.8344	-49.8990
	gic21	-95.51667*	3.70087	.000	-118.4844	-72.5490
	gic28	-97.51667*	3.70087	.000	-120.4844	-74.5490

	10cs1	-30.46667*	3.70087	.000	-53.4344	-7.4990
	10cs3	-41.96667*	3.70087	.000	-64.9344	-18.9990
	10cs7	-70.35000*	3.70087	.000	-93.3177	-47.3823
	10cs21	-77.11667*	3.70087	.000	-100.0844	-54.1490
	10cs28	-99.43333*	3.70087	.000	-122.4010	-76.4656
	30cs1	-46.75000*	3.70087	.000	-69.7177	-23.7823
	30cs3	-63.43333*	3.70087	.000	-86.4010	-40.4656
	30cs7	-71.45000*	3.70087	.000	-94.4177	-48.4823
	30cs21	-72.60000*	3.70087	.000	-95.5677	-49.6323
	30cs28	-83.85000*	3.70087	.000	-106.8177	-60.8823
	50cs1	-55.68333*	3.70087	.000	-78.6510	-32.7156
	50cs3	-44.26667*	3.70087	.000	-67.2344	-21.2990
	50cs7	-65.70000*	3.70087	.000	-88.6677	-42.7323
	50cs21	-49.31667*	3.70087	.000	-72.2844	-26.3490
	50cs28	-49.98333*	3.70087	.000	-72.9510	-27.0156
	mta3	-35.45000*	3.70087	.000	-58.4177	-12.4823
	mta7	-35.46667*	3.70087	.000	-58.4344	-12.4990
	mta21	-83.85000*	3.70087	.000	-106.8177	-60.8823
	mta28	-91.35000*	3.70087	.000	-114.3177	-68.3823
mta3	gic1	-.28333	3.70087	1.000	-23.2510	22.6844
	gic3	-18.23333	3.70087	.457	-41.2010	4.7344
	gic7	-37.41667*	3.70087	.000	-60.3844	-14.4490
	gic21	-60.06667*	3.70087	.000	-83.0344	-37.0990
	gic28	-62.06667*	3.70087	.000	-85.0344	-39.0990
	10cs1	4.98333	3.70087	1.000	-17.9844	27.9510
	10cs3	-6.51667	3.70087	1.000	-29.4844	16.4510
	10cs7	-34.90000*	3.70087	.000	-57.8677	-11.9323
	10cs21	-41.66667*	3.70087	.000	-64.6344	-18.6990
	10cs28	-63.98333*	3.70087	.000	-86.9510	-41.0156
	30cs1	-11.30000	3.70087	.995	-34.2677	11.6677
	30cs3	-27.98333*	3.70087	.001	-50.9510	-5.0156
	30cs7	-36.00000*	3.70087	.000	-58.9677	-13.0323
	30cs21	-37.15000*	3.70087	.000	-60.1177	-14.1823
	30cs28	-48.40000*	3.70087	.000	-71.3677	-25.4323
	50cs1	-20.23333	3.70087	.217	-43.2010	2.7344
	50cs3	-8.81667	3.70087	1.000	-31.7844	14.1510
	50cs7	-30.25000*	3.70087	.000	-53.2177	-7.2823

	50cs21	-13.86667	3.70087	.936	-36.8344	9.1010
	50cs28	-14.53333	3.70087	.896	-37.5010	8.4344
	mta1	35.45000*	3.70087	.000	12.4823	58.4177
	mta7	-.01667	3.70087	1.000	-22.9844	22.9510
	mta21	-48.40000*	3.70087	.000	-71.3677	-25.4323
	mta28	-55.90000*	3.70087	.000	-78.8677	-32.9323
mta7	gic1	-.26667	3.70087	1.000	-23.2344	22.7010
	gic3	-18.21667	3.70087	.460	-41.1844	4.7510
	gic7	-37.40000*	3.70087	.000	-60.3677	-14.4323
	gic21	-60.05000*	3.70087	.000	-83.0177	-37.0823
	gic28	-62.05000*	3.70087	.000	-85.0177	-39.0823
	10cs1	5.00000	3.70087	1.000	-17.9677	27.9677
	10cs3	-6.50000	3.70087	1.000	-29.4677	16.4677
	10cs7	-34.88333*	3.70087	.000	-57.8510	-11.9156
	10cs21	-41.65000*	3.70087	.000	-64.6177	-18.6823
	10cs28	-63.96667*	3.70087	.000	-86.9344	-40.9990
	30cs1	-11.28333	3.70087	.996	-34.2510	11.6844
	30cs3	-27.96667*	3.70087	.001	-50.9344	-4.9990
	30cs7	-35.98333*	3.70087	.000	-58.9510	-13.0156
	30cs21	-37.13333*	3.70087	.000	-60.1010	-14.1656
	30cs28	-48.38333*	3.70087	.000	-71.3510	-25.4156
	50cs1	-20.21667	3.70087	.219	-43.1844	2.7510
	50cs3	-8.80000	3.70087	1.000	-31.7677	14.1677
	50cs7	-30.23333*	3.70087	.000	-53.2010	-7.2656
	50cs21	-13.85000	3.70087	.937	-36.8177	9.1177
	50cs28	-14.51667	3.70087	.897	-37.4844	8.4510
	mta1	35.46667*	3.70087	.000	12.4990	58.4344
	mta3	.01667	3.70087	1.000	-22.9510	22.9844
	mta21	-48.38333*	3.70087	.000	-71.3510	-25.4156
	mta28	-55.88333*	3.70087	.000	-78.8510	-32.9156
mta21	gic1	48.11667*	3.70087	.000	25.1490	71.0844
	gic3	30.16667*	3.70087	.000	7.1990	53.1344
	gic7	10.98333	3.70087	.997	-11.9844	33.9510
	gic21	-11.66667	3.70087	.993	-34.6344	11.3010
	gic28	-13.66667	3.70087	.945	-36.6344	9.3010
	10cs1	53.38333*	3.70087	.000	30.4156	76.3510
	10cs3	41.88333*	3.70087	.000	18.9156	64.8510

	10cs7	13.50000	3.70087	.953	-9.4677	36.4677
	10cs21	6.73333	3.70087	1.000	-16.2344	29.7010
	10cs28	-15.58333	3.70087	.803	-38.5510	7.3844
	30cs1	37.10000*	3.70087	.000	14.1323	60.0677
	30cs3	20.41667	3.70087	.200	-2.5510	43.3844
	30cs7	12.40000	3.70087	.983	-10.5677	35.3677
	30cs21	11.25000	3.70087	.996	-11.7177	34.2177
	30cs28	.00000	3.70087	1.000	-22.9677	22.9677
	50cs1	28.16667*	3.70087	.001	5.1990	51.1344
	50cs3	39.58333*	3.70087	.000	16.6156	62.5510
	50cs7	18.15000	3.70087	.469	-4.8177	41.1177
	50cs21	34.53333*	3.70087	.000	11.5656	57.5010
	50cs28	33.86667*	3.70087	.000	10.8990	56.8344
	mta1	83.85000*	3.70087	.000	60.8823	106.8177
	mta3	48.40000*	3.70087	.000	25.4323	71.3677
	mta7	48.38333*	3.70087	.000	25.4156	71.3510
	mta28	-7.50000	3.70087	1.000	-30.4677	15.4677
mta28	gic1	55.61667*	3.70087	.000	32.6490	78.5844
	gic3	37.66667*	3.70087	.000	14.6990	60.6344
	gic7	18.48333	3.70087	.423	-4.4844	41.4510
	gic21	-4.16667	3.70087	1.000	-27.1344	18.8010
	gic28	-6.16667	3.70087	1.000	-29.1344	16.8010
	10cs1	60.88333*	3.70087	.000	37.9156	83.8510
	10cs3	49.38333*	3.70087	.000	26.4156	72.3510
	10cs7	21.00000	3.70087	.151	-1.9677	43.9677
	10cs21	14.23333	3.70087	.915	-8.7344	37.2010
	10cs28	-8.08333	3.70087	1.000	-31.0510	14.8844
	30cs1	44.60000*	3.70087	.000	21.6323	67.5677
	30cs3	27.91667*	3.70087	.001	4.9490	50.8844
	30cs7	19.90000	3.70087	.251	-3.0677	42.8677
	30cs21	18.75000	3.70087	.388	-4.2177	41.7177
	30cs28	7.50000	3.70087	1.000	-15.4677	30.4677
	50cs1	35.66667*	3.70087	.000	12.6990	58.6344
	50cs3	47.08333*	3.70087	.000	24.1156	70.0510
	50cs7	25.65000*	3.70087	.007	2.6823	48.6177
	50cs21	42.03333*	3.70087	.000	19.0656	65.0010

	50cs28	41.36667*	3.70087	.000	18.3990	64.3344
	mta1	91.35000*	3.70087	.000	68.3823	114.3177
	mta3	55.90000*	3.70087	.000	32.9323	78.8677
	mta7	55.88333*	3.70087	.000	32.9156	78.8510
	mta21	7.50000	3.70087	1.000	-15.4677	30.4677

*. The mean difference is significant at the 0.05 level.

Table 16 Homogeneous Subsets for compressive strength

strength

Scheffe[a]

group.time	N	Subset for alpha = 0.05					
		1	2	3	4	5	6
mta1	6	30.9833					
10cs1	6		61.4500				
mta3	6		66.4333	66.4333			
mta7	6		66.4500	66.4500			
gic1	6		66.7167	66.7167			
10cs3	6		72.9500	72.9500	72.9500		
50cs3	6		75.2500	75.2500	75.2500	75.2500	
30cs1	6		77.7333	77.7333	77.7333	77.7333	
50cs21	6		80.3000	80.3000	80.3000	80.3000	80.3000
50cs28	6		80.9667	80.9667	80.9667	80.9667	80.9667
gic3	6			84.6667	84.6667	84.6667	84.6667
50cs1	6			86.6667	86.6667	86.6667	86.6667
30cs3	6				94.4167	94.4167	94.4167
50cs7	6					96.6833	96.6833
10cs7	6						101.3333
30cs7	6						102.4333
30cs21	6						
gic7	6						
10cs21	6						
30cs28	6						
mta21	6						
mta28	6						
gic21	6						
gic28	6						
10cs28	6						
Sig.		1.000	.293	.217	.119	.121	.083

Means for groups in homogeneous subsets are displayed.

a. Uses Harmonic Mean Sample Size = 6.000.

strength

Scheffe[a]

group.time	Subset for alpha = 0.05					
	7	8	9	10	11	12
50cs28	80.9667					
gic3	84.6667					
50cs1	86.6667	86.6667				
30cs3	94.4167	94.4167	94.4167			
50cs7	96.6833	96.6833	96.6833			
10cs7	101.3333	101.3333	101.3333	101.3333		
30cs7	102.4333	102.4333	102.4333	102.4333		
30cs21	103.5833	103.5833	103.5833	103.5833	103.5833	
gic7	103.8500	103.8500	103.8500	103.8500	103.8500	
10cs21		108.1000	108.1000	108.1000	108.1000	108.1000
30cs28			114.8333	114.8333	114.8333	114.8333
mta21			114.8333	114.8333	114.8333	114.8333
mta28				122.3333	122.3333	122.3333
gic21					126.5000	126.5000
gic28						128.5000
10cs28						130.4167
Sig.	.053	.121	.200	.151	.052	.074

Means for groups in homogeneous subsets are displayed.

a. Uses Harmonic Mean Sample Size = 6.000.

VITAE

VITAE

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