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Acidity of Light-Cured Glass Isomer Materials

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ACIDITY OF LIGHT-CURED GLASS IONOMER MATERIALS

BY

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DEDICATION

I dedicate this thesis to my parents;
my husband, Abed and my sons
Saif and Mhanna

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VITA

The author, Najwa Jowharji, is the daughter of Mansour Jowharji and Suad Alyaman. She was born October 16, 1961, in Jeddah, Saudi Arabia.

Her elementary education was obtained in the public schools of Saudi Arabia. Her secondary education was completed in 1979 at the First Secondary School, Taif, Saudi Arabia.

In 1979, Dr. Jowharji entered King Saud University, receiving the degree of Bachelor of dental surgery (B.D.S.) in May, 1985.

Dr. Jowharji was granted a scholarship from her school (King Saud University) in 1985, to study in the united states.

In September, 1985, Dr. Jowharji entered the general residency program at the University of Minnesota, which was completed in one year.

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In June, 1989, Dr. Jowharji was accepted in the graduate School of Loyola University in the Oral Biology Program, enabling her to complete the Master of Science in January 1991.

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INTRODUCTION

Glass ionomers have a wide range of uses in dentistry today. They can serve as fillings, bases, liners, luting cements, cores and fissure sealants. Since their emergence in the early 1970's, they have undergone a tremendous development.

Recently, light-cured glass ionomer liners were introduced to replace the traditional chemical cured glass ionomers. Their major advantages are increased working time, shorter setting time, improved strength and increased acid resistance. [12] However, their acidity has not been studied.

Previous research suggested that pulpal irritation could be caused by the initial acidity of the prolonged chemical reaction of the self cured material. One of the major advantages of the light - cured glass ionomer liners is the setting reaction will be triggered by visible light which shorten the setting time (20-30 sec).

The purpose of the present study was:

1. to investigate in vitro the pH during setting of four types of light-cured glass ionomer liners.
2. to compare the pH of light cured glass ionomers with six self cured glass ionomers.
3. to compare the pH of the glass ionomer materials with widely used zinc polycarboxylate and zinc phosphate cements.

LITERATURE REVIEW

Development of glass-ionomer cements

The glass-ionomer cements are a new and interesting development in adhesive dentistry. This dental cement system were developed in 1971 by Wilson and Kent [1] which is based on the hardening reaction between an ion-leachable aluminosilicate glass and aqueous solutions of polymers and co-polymers of acrylic acid (ASPA). The intention is to develop this material for a variety of dental applications such as the restoration of anterior teeth, the filling of erosion cavities, general cementation and cavity linings.[2]

In 1973, Kent et al. found a glass that was high in fluoride (G-200) [*a] and mixed with 50 percent aqueous solution of polyacrylic acid which gave a useable cement, ASPA I. [3,4] The properties of the ASPA cement were compared with those of existing dental cement. It appears to combine certain favorable properties of dental silicate and polycarboxylate cements. [3] However, one of the problem associated with ASPA I, is the limited working time and the slow rate of surface hardening. This has been improved by adding chelating agents such a tartaric acid which increase the rate of hardening without reducing the working time. [5] This refinement of ASPA I was termed ASPA II and constituted the first practical glass-ionomer cement. Even by today's standards its properties were excellent.[3]

*a. G-200 is a designation of the Laboratory of the Government Chemist (LGC) (London)

Attempts to improve the reactivity of glass powder by increasing the AL_2O_3 :SiO_2 ratio have been also reported by Kent, Lewis and Wilson. [4] This discovery enabled more reactive glasses to be prepared suitable for forming rapid setting cement with polyacrylic acid which is a weaker acid than phosphoric acid used in dental silicate cement.

Problems associated with the use of polyacrylic acid have been reported. Its viscosity was high and the liquid tends to gel. This problem was solved by Crisp and Wilson [6], who developed a copolymer of acrylic and itaconic acid that did not gel at 50% concentrations in aqueous solution. In this form glass ionomer cement was termed ASPA IV and was considered suitable for commercial production as a fissure filling material and for treatment of erosion cavities. [7,8]

Studies have been made to combine the desirable properties of silicate cements, composite and polycarboxylate cements which have been achieved in the developments of the glass ionomer cement system. These new cements designed for a number of specific clinical applications. [8,9,10] The biological compatibility, effective maximum grain size, retentive ability, disintegration in and absorption of water and solubility in acid proved to be fully acceptable.[11]

Recently, light-cured glass ionomer liners were introduced. Their major advantages are increased working time, shorter setting time, improved strength and increased acid resistance. [12]

Glass Composition

Cement properties depend on chemical composition, particle-size distribution of the powder, molecular weight and concentration of the liquid polyacid. [3] The powder of a glass ionomer cement is a calcium fluoro-

aluminosilicate glass with a formula of SiO_2-Al_2O_3-CaF_2-Na_3AlF_6-$AlPO_4$. The nominal composition of the glass is listed in table 1. [13,14]

Investigations carried out on variants of these glasses showed that their reactivity depended on the ratio of alumina to silica in the fusion mixture used for their preparation. This ratio, which is the ratio of a basic oxide to an acidic oxide, determines the basicity of the glass. Because the reaction between glass and liquid is an acid base one, an increase in the basicity of the glass will increase the rate of setting reaction. [2] According to Wilson and McLean, [15] the Al_2O_3/SiO_2 ratio is required to be 1:2 or more and the fluoride content can be up to 23%. The visual appearance of the glass could be clear, opal or opaque depending on its chemical composition. Glasses high in calcium fluoride or alumina are opaque. This opaqueness arises from the presence of dispersed crystalline phases of fluoride or corundum. The addition of cryolite (Na_3AlF_6) reduces the temperature at which the glass will fuse and increases the translucency of the set cement. Aluminum phosphate is added to improves the translucency and to add body to the cement paste.

Liquid Composition

The liquid typically is a 47.5% solution of 2:1 polyacrylic acid/itaconic acid copolymer (average molecular weight 10,000) in water. [14] The copolymer may also be freeze-dried and incorporated into the powder. In addition to the acrylic acid-itaconic acid copolymer, it also contains a small amount of tartaric acid, in the range of 5%. The itaconic acid reduces the viscosity of the liquid and inhibits gelation. The tartaric acid can be added to improve the working and setting characteristics. [5,6,16,17]

Table 1

Nominal composition of calcium fluoroalumino silicate glass used in powder of glass ionomer cement. [13]

Chemical	Percent by weight
SiO_2	29.0
Al_2O_3	16.6
CaF_2	34.3
$Na\ AlF_6$	5.0
AlF_3	5.3
$AlPO_4$	9.9

Polyacrylic acid is a weaker acid than phosphoric acid and a more basic glass is required to produce equivalent setting, hence the proportion of alumina to silica has to be greater.[2] When polyacrylic acid is dissociated, hydrogen ions tends to be bonded to the polyelectrolyte chain and the large polyacrylic molecules will show less tendency to diffuse along dentinal tubules than the smaller phosphoric acid molecules. In addition, with a long chain polyacid containing a multiplicity of functional groups, ion binding at only one of these to the bulk of the cement will tend to hinder its migration. [2]

Recently, four light-cured glass ionomer systems have become commercially available.[12,39] In Vitrabond liner/base, the powder contains a fluoroaluminosilicate glass and some of the chemical components of the light activated resin accelerator. The liquid contains a polyacrylic acid copolymer with pendant methacrylate groups, 25% HEMA (hydroxyethylmethacrylate), additional photo accelerators, and water. After mixing the resulting material contains 10% HEMA.

The second system called XR-Ionomer (Kerr, Manufacturing Co.), the powder is a calcium aluminofluorosilicate glass and the liquid is polyacrylic acid with pendent methacrylate groups. The XR-Ionomer liquid differs from Vitrabond's in that its polyacrylic acid contains about half the number of pendant methacrylate groups. In addition there is no HEMA in the liquid.

The third system called TimeLine (L.D. Caulk), is not a glass ionomer system. Its a one-part material of medium viscosity containing a relatively hydrophobic dimethacrylate resin matrix, filled with radiopaque glass and sodium fluoride powder. It has an initial fluoride release (20 ppm). However, this drops to 40% of the release of a conventional glass ionomer liner after 1 year. An

additional brand of glass ionomer, Zionomer (DentMat), was used in this study, however, no published data on its composition exist in the literature at this time.

Chemistry and setting reaction of glass ionomer cement

Chemical studies on the reaction of the glass ionomer cement showed that the setting mechanism is an acid base reaction between the acidic polyelectrolyte and the alumino silicate glass. [14,16,19] The setting reaction of glass ionomer cement is reported to take place in several overlapping stages.[10,20]

In a freshly mixed paste, its presumed that hydrated protons from the liquid penetrate the surface regions of the powder particles, displacing cations (Al^{3+}, Ca^{2+}) and degrading the alumino silicate network into the aqueous phase of the cement paste. Metallic salt bridges are then formed between the long chains of charged polycarboxylate ions, cross linking them and causing the aqueous phase to gel and the cement to set to an amorphous mass. [16,19,20]

At the first stage of the reaction calcium ions are more rapidly bound to the polyacrylate chains than aluminum ions and are chiefly responsible for its initial set. [2,16,20] At the second stage of the reaction, the aluminum salt is formed and it is responsible for the final hardening of glass ionomer cement. In this stage the cement shows considerable increase in hardness and stiffness as well as resistance to plastic deformation. [21,22,23] McLean and Wilson stated that the cement initially sets to a condition which enables it to be carved like an amalgam (calcium ion-exchange), later it sets rock hard (aluminium ion-exchange). [7] The fluoride and phosphate ions form insoluble salts and complexes. The sodium ions form a silica gel. The structure of the fully set cement is a composite of glass particles surrounded by silica gel in a matrix of polyanions cross-linked by ionic bridges.

Within the matrix are small particles of silica gel containing fluorite crystallites. [14]

Effects on pulp tissue

The dentin and the pulp must be considered as one organ (the pulp-dentin complex) because of the intimate relationship between the cellular tissue within the dentin and the peripheral pulp tissue. The dentinal tubules occupy from 20%-39% of dentin, and the dentinal fluid within represents about 22% of the total volume of dentin. [24,25,26]

Reports as to the cause of pulpal irritation from glass ionomer cements have fluctuated between the initial acidity of the material and the influence of bacteria. Brannstrom in 1984 reported that the pulpal inflammation may arise from bacterial infection rather than from the filling material or the pre-treatment procedures. [27] In another study, however, Plant, et. al showed no correlation between pulpal inflammation and microleakage of all glass ionomer cements tested. Upon histological examination all pulps in teeth filled with glass ionomer cements revealed some degree of inflammation. [28]

In a report to the American Dental Association's Council on Dental Materials, Instruments, and Equipment it was noted that sensitivity and pulpal death occurred in some cases when glass ionomers were used for crown cementation as a luting agent. [29] This hypersensitivity was explained by Gunilla and Brannstrom who indicated that some materials are hygroscopic and may dehydrate dentin producing centrifugal flow of fluid in the dentinal tubules. This dehydration of dentin which may elicit pain and result in aspiration of odontoblasts into tubule. [30] A luting mix has a higher toxicity than a thick base mix and after a four days a tremendous number of neutrophils have been found to infiltrate the pulp tissue. [31]

To investigate the pulp response a clinical study by Norman and Wright compared the responses of patients to a glass ionomer cement (Ketac-Cem) and zinc phosphate cement (Tenacin) used in cementation of various types of castings. They concluded that, after six months, both cements produced similar pulpal response and either cement can be used safely for crown cementation. They also indicated that bacterial or marginal leakage can induce hypersensitivity[32]. This finding has been studied by Hey's et.al using different types of glass ionomers and zinc phosphate cement (Tenacin) in Rhesus monkeys. They found that hypersensitivity after crown cementation did not result from bacteria or marginal leakage. Since evaluation of the pulp response was not statistically significant, they concluded that other factors may contribute to hypersensitivity after crown cementation. [33]

In order to distinguish material toxicity from bacterial effect, Patterson and Watts examined ASPA (De-Trey) by placing it directly on exposed dental pulps of germ free rat molars. They found a localized zone of pulpal necrosis with inhibition of calcific repair. [34]

A human histological study showed evidence of severe pulpal response beneath glass ionomer compared to zinc oxide-eugenol, and a significant positive correlation was found between pulpal inflammation and bacterial leakage. [35] In another study using monkeys, evaluation of pulpal response showed no significant difference between glass ionomer cement and zinc oxide-eugenol. [36] Additionally, the culture tissue study showed less cytotoxic action than zinc oxide-eugenol.

Other varying degrees of toxicity have been described when glass ionomer cements were placed in tissue culture. Hume and Mount reported that each of the

tested glass ionomer cements was severely toxic. This finding supports the proposal that acid release may be a factor contributing to the observed cytotoxicity.[37] The pH and the amount of the free acid depend on the setting rate of the cement. [31] This also was a concern when Smith and Ruse suggested that the initial acidity of glass ionomer cements may contribute to their damaging effect on the pulp. [38]

Light-cured glass ionomer liners were introduced in late 1989, at this time very little material has been reported in the literature. Some of the chemical and physical properties of three new types of light cured glass ionomer (TimeLine, Vitrabond, and XR-Ionomer) have been reported. Light-cured glass ionomer had a lower acidity and a setting time of 20-30 seconds compared with 4.5-5.0 minutes for conventional types. [12,39]

MATERIALS AND METHODS

Materials Investigated

Four commercially available light-cured and six self-cured glass ionomers were used in this study. Two zinc polycarboxylate and zinc phosphate cements were also investigated in this study for comparison and as control groups(table 2).

The materials selected represent the leading brands on the markets. Light cured glass ionomers, zinc phosphate cement are American products. GC Fuji I, GC-Dentin cement, shofu glass ionomer and shofu polycarboxylate are Japanese products. Ketac-cem, Katac-Bond, Durelon and BaseLine represent the European products.

Details of the chemical composition, mode of supply, methods of polymerization, and manufacturer are presented in table 2.

Assembly and specimens preparation

The pH of the tested materials were measured using the following assembly(Fig.1)

1. A corning module 10 pH meter [a].
2. pH electrode (flat surface polymer body combination electrode) [a].
3. A standardized metal stand to hold the electrode at fixed distance from the sample surface each time.

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*a. Corning Medical and Scientific, Corning Glass Works. Medfield, MA 02052 USA.

Table 2
Glass Ionomer Cements, Liner/Base Investigated

No.	Materials	Cure	Mode of supply	code	Batch No.	Manufacturer
1	vitra bond	light	liquid/powder	VB	7510	3M Company, St. Paul, MN 55414
2	TimeLine	light	paste	TL	012389	Caulk Company Milford, DW 19963
3	XR-Ionomer	light	liquid/powder	XR	3606 -21626	Kerr Company Romulus, MI 48174
4	Zionomer	light	liquid/powder	ZI	powder 498013 liquid 499008	Den-Mat Corp. Santa Maria, CA 93456
5	Ketac-Cem	chemical	liquid/powder	KC	021787	ESPE-premier praparate GMBH Co.KG D-8031 seefeld/oberbay
6	GC Fuji I	chemical	liquid/powder	FI	210971	G-C Industrial Corp. Tokyo, Japan
7	Shofu Type I	chemical	liquid/powder	SG	082086	Shofu Dental Corp. Menlo Park, CA 94025
8	Katac-Bond	chemical	liquid/powder	KB	080486	ESPE-premier praparate GMBH Co.KG D-8031 seefeld/oberbay
9	BaseLine	chemical	liquid/powder (water)	BL	890181	DeTrey-Dentsphy Weybridge,Surrey,England
10	GC Dentin cement	chemical	liquid/powder	DC	080592	G-C Industrial Corp. Tokyo, Japan
11	Shofu Hy-Bond polycarboxylate	chemical	liquid/powder	SP	103086	Shofu Dental Corp. Menlo Park, CA 94025
12	Durelon	chemical	liquid/powder	DP	0135	ESPE praparate GMBH Co.KG D-8031 seefeld/oberbay
13	Zinc phosphate	chemical	liquid/powder	ZP	0208710	Mission White Dental,INC Tinton Falls, NJ 07724

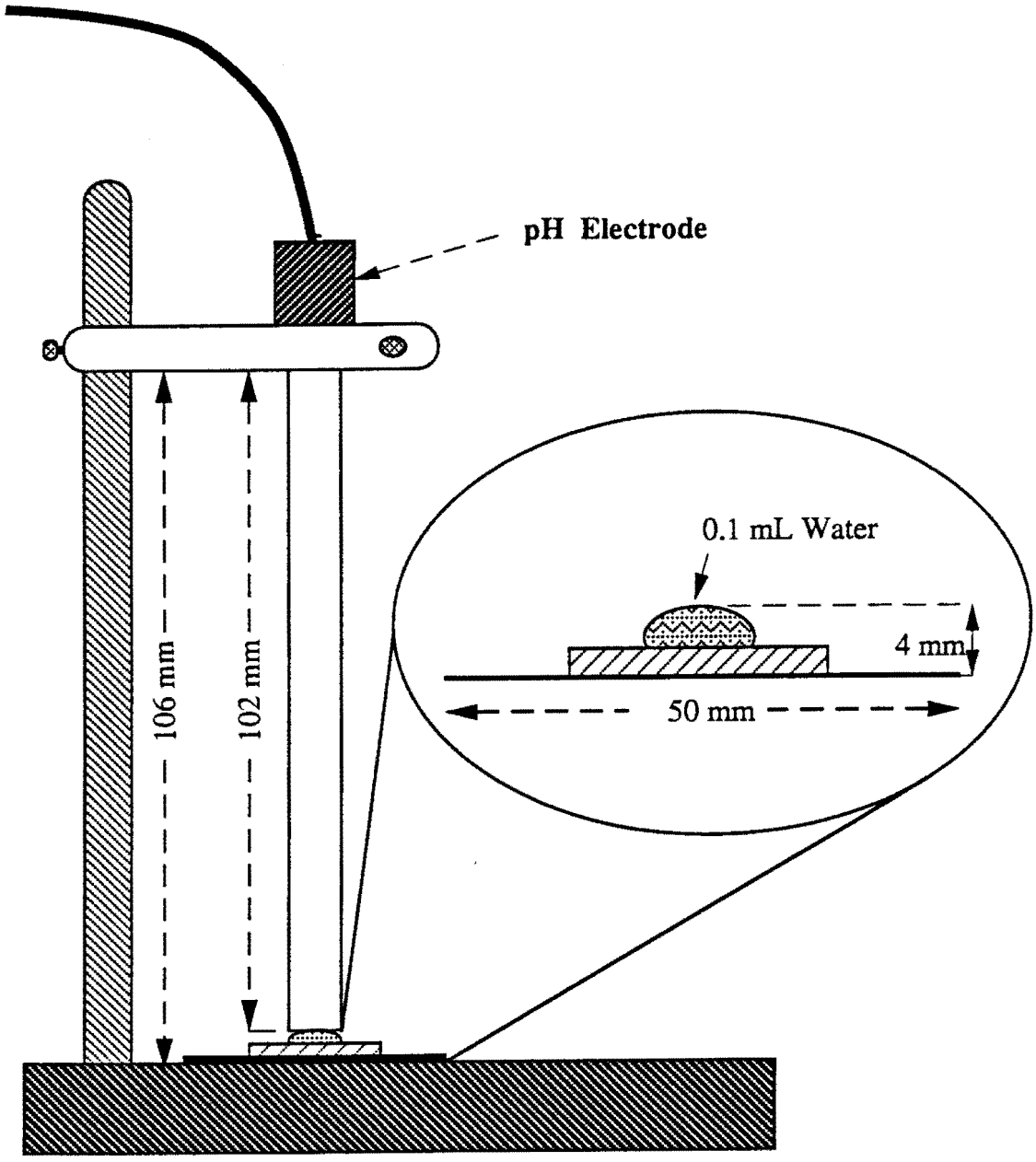


Figure 1

Diagrammatic illustration of a specimen placed on the stand under the pH electrode

Sample dimensions were made using a standardized metal ring measuring 20mm. in diameter and 1mm. in thickness, ADA specification No. 27,4.3.6. Two square glass plate 5 x 5 cm and 2 mm thick, two square myler plastic sheets .0635 mm thick and two binder clips were also used to make the samples (fig 2).

Five specimens of each material were dispensed accurately according to the respective manufacturer's instructions and mixed under room conditions (22-23°C and 30% to 50% relative humidity). (table 3)

After mixing, the cement was immediately placed in the metal ring. In making specimens, the cement-filled metal ring was pressed between the mylar sheets and two glass plates to extrude the excess cement and to insure parallel and smooth surfaces by means of the two metal paper clips. If its a light cured material, the sample was cured for 20-30 second using the same light activating machine each time.

The specimens were placed in a humidity chamber at 37°C and 80% relative humidity. At the time of measurement each specimen was removed from the humidity chamber and placed on the stand at room temperature and a two drops of deionized water (.1ml) were placed on the surface of the set cement.
Before taking any measurements, the pH meter was calibrated by using a standard pH 4 buffer solution (potassium acid phthalate). The electrode was then placed to contact the water at a fixed distance and the reading was recorded.

This procedure was repeated after 15, 30, 45, 60, 120, 180 minutes and after 24 hours from mixing time. Between measurements, the electrode was cleaned, recalibrated and stored in a potassium acid phthalate, pH 4.

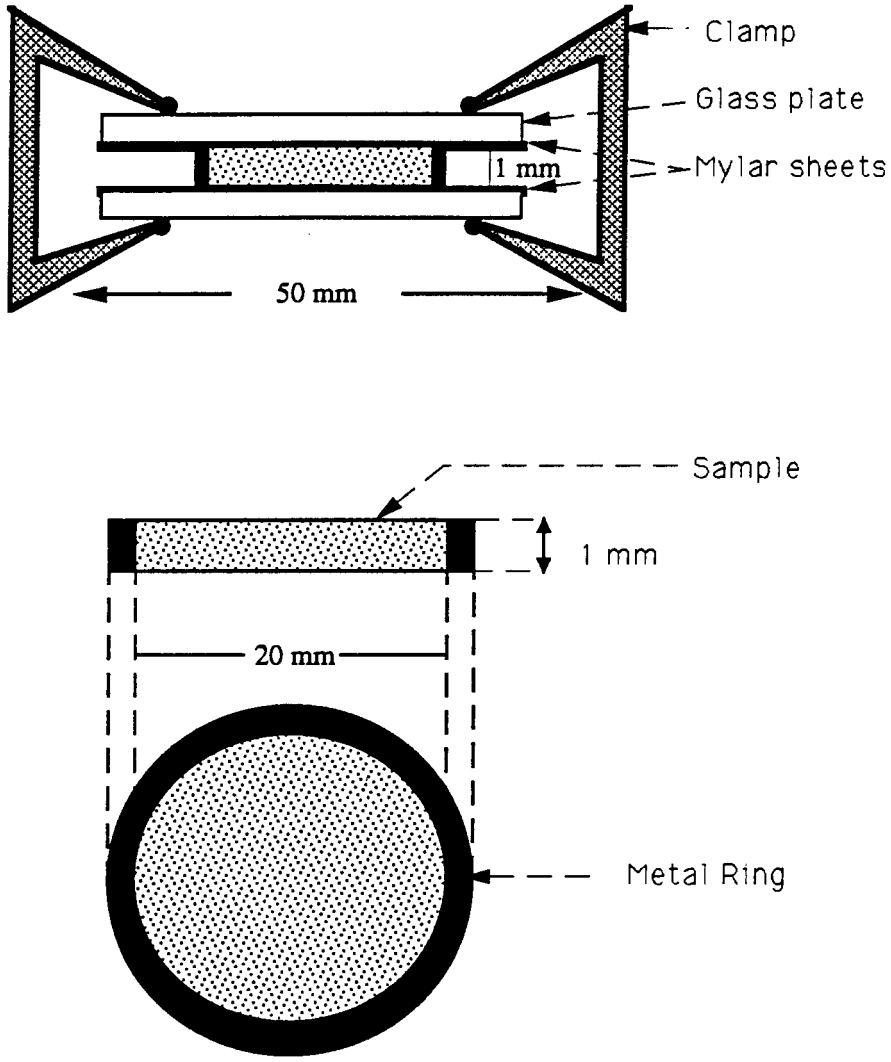


Figure 2

Diagrammatic illustration of the assembly used to prepare specimens

Table 3
Powder-Liquid Ratio Required by Manufacturer

Materials	Mode	Powder (scoop)	Liquid (drops)
Vitrabond	Base/liner	1	1
Timeline	Base/liner	single part paste	
XR-Ionomer	Base/liner	1 capsule	2
Zionomer	Base/liner	2	3
Katac-Cem	Base	2	3
GC Fuji I	Luting	1	2
Shofu Type I	Liner	2	3
Ketac-Bond	Base	1	1
Base Line	Base	2	2 (water)
GC Dentin Cement	Base	1	1
Hy-Bond polycarboxylate	Luting	1	3
Durelon	Base	1	2
Zinc phosphate	Base	1 scoop from large well and 1 scoop from small well	3

Statistical Methodology

A two way analysis of variance (ANOVA) was performed to test the difference between the acidity level for all materials stored for seven different times. Whenever the two-way (ANOVA) revealed a significant time by material interaction difference, a one way ANOVA was performed for each material, to test the effect of storage times and at each storage time to compare between materials. In all cases for which the one-way ANOVA showed an overall difference between means, a Schaff'e test was used to compare all possible pairs of means at ($p = .01$ level).

RESULTS

A summary of mean pH values, standard deviations, and number of specimens are presented in Table 4 for light-cured glass ionomers, polycarboxylates and zinc phosphate cement; and in Table 5 for self-cured glass ionomer materials. Mean values ± standard deviation are also graphically presented in the Appendix Figures 6 through 9.

Light-cured glass ionomer materials shows a minimum mean range of pH (4.52 ± 0.16) to (5.47 ± 0.02) for XR-Ionomer and Zionomer, respectively at 15 minutes from mixing time. A maximum mean range of pH (5.57 ± 0.17) to (6.72 ± 0.08) for Vitrabond and Zionomer, respectively after 24 hours of storage time.

Self-cured glass ionomer materials shows a minimum mean range of pH ± standard deviation (3.65 ± 0.27) to (4.79 ± 0.66) for BaseLine, and Shofu Type I glass ionomer, respectively at 15 minutes storage time. A maximum range of (4.70 ± 0.22) to (6.47 ± 0.04) is showed for BaseLine and Ketac-Bond, respectively at 24 hours storage time.

Polycarboxylate materials (Shofu Hy-Bond and Durelon) show a minimum of pH mean ± standard deviation of (4.32 ± 0.52, 4.57 ± 0.11) at 15 minutes storage time and a maximum of (6.5 ± 0.17, 6.3 ± 0.19) at 24 hours storage time respectively. Zinc phosphate cement, shows a minimum of (4.31 ± 0.33) at 15 minutes and a maximum of (6.18 ± 0.72) at 24 hours storage time.

Table 4

Mean pH values (x), standard deviation (s.d.) and number (N) of specimen for light-cured glass ionomers and control groups.

		Storage Times In Minutes						
Materials	**Statistics**	**15**	**30**	**45**	**60**	**120**	**180**	**1440**
VB	X	5.21	5.21	5.37	5.50	5.48	5.28	5.57
	s.d.	0.33	0.34	0.35	0.12	0.14	0.63	0.17
	N	5	5	5	5	5	5	5
TL	X	5.4	6.01	5.99	6.10	6.15	6.22	6.23
	s.d.	0.31	0.11	0.22	0.10	0.09	0.07	0.10
	N	5	5	5	5	5	5	5
XR	X	4.52	4.94	5.16	5.40	5.79	6.21	6.48
	s.d.	0.16	0.14	0.34	0.31	0.65	0.14	0.16
	N	5	5	5	5	5	5	5
ZI	X	5.47	6.08	6.22	6.02	6.26	6.43	6.72
	s.d.	0.02	0.15	0.02	0.19	0.11	0.08	0.08
	N	5	5	5	5	5	5	5
SP	X	4.32	5.20	5.81	6.06	6.09	5.95	6.50
	s.d.	0.52	0.60	0.18	0.13	0.09	0.22	0.17
	N	5	5	5	5	5	5	5
DP	X	4.57	5.96	6.20	6.21	5.74	5.82	6.30
	s.d.	0.11	0.35	0.01	0.05	0.08	0.06	0.19
	N	5	5	5	5	5	5	5
ZP	X	4.31	4.86	5.15	5.19	5.16	5.48	6.18
	s.d.	0.33	0.16	0.14	0.18	0.16	0.26	0.72
	N	5	5	5	5	5	5	5

VB = Vitrabond

TL = TimeLine

XR = XR Ionomer

ZI = Zionomer

Sp = Shofu Hy-Bond polycarboxylate

Dp = Durelon

Zp = Zinc phosphate

Table 5

Mean pH values (x), Standard deviation (s.d.) and number (N) of specimen for self-cured glass ionomers.

		Storage Times In Minutes						
Materials	**Statistics**	**15**	**30**	**45**	**60**	**120**	**180**	**1440**
KC	X	4.74	5.51	5.47	5.62	5.87	5.86	6.32
	s.d.	0.38	0.36	0.20	0.13	0.11	0.09	0.33
	N	5	5	5	5	5	5	5
FI	X	3.71	4.42	4.73	4.76	4.19	4.70	5.95
	s.d.	0.20	0.32	0.44	0.27	0.56	0.33	0.59
	N	5	5	5	5	5	5	5
SG	X	4.79	5.65	6.00	6.05	5.58	5.90	6.23
	s.d.	0.66	0.37	0.21	0.16	0.37	0.42	0.23
	N	5	5	5	5	5	5	5
KB	X	4.43	5.01	5.49	5.58	5.20	5.31	6.47
	s.d.	0.46	0.52	0.09	0.03	0.13	0.02	0.04
	N	5	5	5	5	5	5	5
BL	X	3.65	4.47	4.84	4.95	4.35	4.41	4.70
	s.d.	0.27	0.34	0.20	0.07	0.27	0.18	0.22
	N	5	5	5	5	5	5	5
DC	X	4.68	5.09	5.54	5.81	5.94	5.98	6.39
	s.d.	0.29	0.26	0.20	0.25	0.26	0.27	0.17
	N	5	5	5	5	5	5	5

KC = Ketac-Cem KB = Ketac-Bond

FI = GC Fuji Ionomer BL = BaseLine

SG = Shofu Type I DC = Dentin cement

Two-Way Analysis of Variance

A two way analysis of variance (ANOVA) to test the effect of storage time, material, and their interaction was performed. The analysis revealed a significant material-by-time interaction ($p = 0.0001$) at alpha nominal level = 0.01, as shown in Table 19 in the Appendix. The overall effect of time or the overall effect of material could not be assessed; therefore a one way analysis of variance was performed to compare the difference between materials at each storage time, and to evaluate the storage times effect on each material.

Material Effect

Values for material specific pH means at each storage time, are presented in Figure 6 in the Appendix. To evaluate the difference between materials at each storage time, a one-way analysis of variance at 1% nominal level is performed between:

A - Light-cured glass ionomer materials

B - Self-cured glass ionomer materials

C - All tested materials

One-Way Analysis of Variance Between Light-Cured Materials

Results of the one way ANOVA, between light-cured glass ionomer materials, Tables 20 through 26 in the Appendix revealed a highly significant difference between materials at all storage times ($P \leq 0.0003$) except at 120 storage time where there is no significant difference with $p = 0.157$. Results of the Scheff'e specific comparison between means, at the 1 percent nominal level and 15 minutes storage time (Table 6) indicate the significant differences between the low pH

values of XR-Ionomer (4.52 ± 0.16) vs TimeLine (5.4 ± 0.31) and Zionomer (5.47 ± 0.02). After 24 hours storage time, Scheff'e specific comparison, (Table 7), indicates significant differences are between:

* Vitrabond vs. TimeLine, XR-Ionomer and Zionomer.
* TimeLine vs. Zionomer

Scheff'e specific comparison between means also performed for other test times and the results are summarized in, Table 8.

Comparing Light-Cured with Control Groups

To compare light-cured glass ionomers with control groups (Shofu polycarboxylate, Durelon and Zinc phosphate) another one-way ANOVA was performed at each storage time and at 1 percent alpha level. ANOVA Tables are listed in the Appendix Table 27 through 33. The analysis revealed a highly significant difference between light cured glass ionomers and control groups ($p \leq 0.0002$). Results of Scheff'e test between means, at 1 percent nominal level, indicate the differences are caused by the low mean pH value of all control groups vs. all light-cured glass ionomer except for XR-Ionomer, at 15 minutes storage time, Table 9. At 24 hours storage time, Scheff'e test, Table 10, shows that the significant difference is between the low mean pH values of Vitrabond (5.57 ± 0.17) vs the highest mean pH values of Shofu Hy-Bond polycarboxylate, (6.5 ± 0.17).

Scheff'e test also performed at 1 percent nominal level, at the other storage times, and results are presented in Table 11.

Table 6

Matrixes of Scheff'e multiple comparison tests between Light-cured Glass Ionomer Materials at 15 minutes storage time.

(Stars indicate significant difference at 1% Alpha level)

	VB	TL	XR	ZI
VB		--	--	--
TL			*	--
XR				*

Table 7

Matrixes of Scheff'e multiple comparison tests between Light-cured Glass Ionomer Materials at 24 hours storage time.

(Stars indicate significant difference at 1% Alpha level).

	VB	TL	XR	ZI
VB		*	*	*
TL			--	*
XR				--

Table 8

Matrixes of Scheff'e multiple comparison tests between Light-cured Glass Ionomer Materials. (Stars indicate significant difference at 1% Alpha level).

Storage Times In Minutes		VB	TL	XR	ZI
30	VB		*	--	*
	TL			*	--
	XR				*
45	VB		--	--	*
	TL			*	--
	XR				*
60	VB		--	--	--
	TL			--	--
	XR				--
120	VB		*	--	--
	TL			*	--
	XR				*
180	VB		--	--	*
	TL			--	--
	XR				--

Table 9

Matrixes of Scheff'e multiple comparison tests between Light-cured Glass Ionomers and Control Materials at 15 minutes storage times.

(Stars indicate significant difference at 1% Alpha level).

	VB	TL	XR	ZI	SP	DP	ZP
VB		--	--	--	*	--	*
TL			*	--	*	*	*
XR				*	--	--	--
ZI					*	*	*
SP						--	--
DP							--

Table 10

Matrixes of Scheff'e multiple comparison tests between Light-cured Glass Ionomers And Control Materials at 24 hours storage time.

(Stars indicate significant difference at 1% Alpha level).

	VB	TL	XR	ZI	SP	DP	ZP
VB		--	--	*	*	--	--
TL			--	--	--	--	--
XR				--	--	--	--
ZI					--	--	--
SP						--	--
DP							--

Table 11

Matrixes of Scheff'e multiple comparison tests between Light-cured Glass Ionomers and Control Materials. (Stars indicate significant difference at 1% Alpha level).

Storage Times In Minutes		VB	TL	XR	ZI	SP	DP	ZP
30	VB		*	--	*	--	*	--
	TL			*	--	*	--	*
	XR				*	--	*	--
	ZI					*	--	*
	SP						*	--
	DP							*
45	VB		--	--	*	--	*	--
	TL			*	--	--	--	*
	XR				*	*	*	--
	ZI					--	--	*
	SP						--	*
	DP							*
60	VB		*	--	--	*	*	--
	TL			*	--	--	--	*
	XR				*	*	*	--
	ZI					--	--	*
	SP						--	*
	DP							*
120	VB		--	--	--	--	--	--
	TL			--	--	--	--	*
	XR				--	--	--	--
	ZI					--	--	*
	SP						--	*
	DP							--
180	VB		*	*	*	--	--	--
	TL			--	--	--	--	--
	XR				--	--	--	--
	ZI					--	--	*
	SP						--	--
	DP							--

One-Way Analysis of Variance Between Self-Cured Materials

Results of the one-way ANOVA between self-cured glass ionomer materials are presented in the Appendix Tables 34 through 40. The analysis shows a highly significant difference between materials at all storage times ($p = 0.0001$), except at 15 minutes storage time with p value = 0.0004. Scheff'e specific comparison between materials at 1 percent nominal level and 24 hours storage time (Table 12), indicate the significant difference is caused by the low mean value of BaseLine (4.7 ± 0.22) vs. each of the following: Ketac-Bond (6.47 ± 0.04) Dentin cement (6.39 ± 6.17), Ketac-Cem (6.32 ± 0.33) and Shofu glass ionomer Type I (6.23 ± 0.23).

Scheff'e specific comparison test was also performed for other storage times at the 1 percent nominal level. Results are presented in Table 13.

Comparing Self-Cured with Control Group

In order to compare self-cured glass ionomer with control groups (Shofu Hy-Bond, Durelon and Zinc phosphate) another seven one-way ANOVA was performed at 1 percent alpha level, ANOVA Tables are presented in appendix Tables 41 through 47. The analysis revealed a highly significant difference between materials $p = 0.0001$ at all times. The Scheff'e test indicates, at 15 minutes storage time, the significant difference is between BaseLine vs. Shofu glass ionomer Type I. It also indicates that there is no significant difference between self-cured glass ionomers and control groups. At 24 hours storage time (Table 14) the test shows that the significant difference is caused only by the low mean pH values of BaseLine (4.7 ± 0.22) vs. all self-cured glass ionomers and all control groups.

Table 12

Matrixes of Scheff'e multiple comparison tests between Self-cured Glass Ionomer Materials after 24 hours storage time.

(Stars indicate significant difference at 1% Alpha level).

	KC	FI	SG	KB	BL	DC
KC		--	--	--	*	--
FI			--	--	--	--
SG				--	*	--
KB					*	--
BL						*

Table 13

Matrixes of Scheff'e multiple comparison tests between Self-cured Glass Ionomer Materials. (Stars indicate significant difference at 1% Alpha level).

Storage Times In Minutes		KC	FI	SG	KB	BL	DC
30	KC		*	--	--	--	--
	FI			*	--	--	--
	SG				*	--	--
	KB					--	--
	BL						--
45	KC		--	--	--	--	--
	FI			*	*	--	*
	SG				--	*	--
	KB					--	--
	BL						--
60	KC		*	--	--	*	--
	FI			*	*	--	*
	SG				*	*	--
	KB					*	--
	BL						*
120	KC		*	--	--	*	--
	FI			*	*	--	*
	SG				--	*	--
	KB					--	--
	BL						--
180	KC		*	--	--	*	--
	FI			*	--	--	*
	SG				--	*	--
	KB					*	--
	BL						*

Table 14

Matrixes of Scheff'e multiple comparison tests for Self-cured Glass Ionomers and Control Materials after 24 hours.

(Stars indicate significant difference at 1% Alpha level).

	KC	FI	SG	KB	BL	DC	SP	DP	ZP
KC		--	--	--	*	--	--	--	--
FI			--	--	*	--	--	--	--
SG				--	*	--	--	--	--
KB					*	--	--	--	--
BL						*	*	*	*
DC							--	--	--
SP								--	--
DP									--

Scheff'e test was also performed for other storage times, at 1 percent nominal level, and results are tabulated in Table 15a and 15b.

Comparison Between All Materials

The overall one-way ANOVA for four light-cured glass ionomers, six self-cured glass ionomers, two polycarboxylates and zinc phosphate cements are presented in the Appendix Table 48 through 54. Results show a highly significant difference between materials at all storage times ($p = 0.0001$). Results of the Scheff'e multiple comparison at 1 percent nominal level for 15 minutes and 24 hours storage times are as the following:

1. At 15 minutes storage time (Table 16) the significant difference was caused by the low mean values of self-cured Fuji I (3.71 ± 0.2) and BaseLine (3.65 ± 0.27) vs. each of the light-cured, Zionomer (5.47 ± 0.2), TimeLine (5.4 ± 0.3) and VitraBond (5.21 ± 0.33).

2. At 24 hours storage time (Table 17) the significant difference is mainly caused by the low mean pH values of self-cured BaseLine (4.7 ± 0.22) vs each of the following:

		X ± s.d.
	Fuji I	(5.95 ± 0.59)
	Shofu Type I	(6.23 ± 0.23)
self-cured	Ketac-cem	(6.32 ± 0.33)
	Dentin-cement	(6.39 ± 0.17)
	Ketac-Bond	(6.47 ± 0.04)

Table 15-a

Matrixes of Scheff'e multiple comparison tests between Self-cured Glass Ionomer and Control Materials for 15, 30 and 45 minutes .

(Stars indicate significant difference at 1% Alpha level).

Storage Times In Minutes		KC	FI	SG	KB	BL	DC	SP	DP	ZP
15	KC		--	--	--	--	--	--	--	--
	FI			--	--	--	--	--	--	--
	SG				--	--	--	--	--	--
	KB					--	--	--	--	--
	BL						--	--	--	--
	DC							--	--	--
	SP								--	--
	DP									--
30	KC		--	--	--	--	--	--	--	--
	FI			*	--	--	--	--	*	--
	SG				--	*	--	--	--	--
	KB					--	--	--	--	--
	BL						--	--	*	--
	DC							--	--	--
	SP								--	--
	DP									--
45	KC		*	--	--	--	--	--	*	--
	FI			*	*	--	*	*	*	--
	SG				--	*	--	--	--	*
	KB					--	--	--	*	--
	BL						*	*	*	--
	DC							--	--	--
	SP								--	--
	DP									*

Table 15-b

Matrixes of Scheff'e multiple comparison tests between Self-cured Glass Ionomer and Control Materials for 60, 120 and 180 minutes.

(Stars indicate significant difference at 1% Alpha level).

Storage Times In Minutes		KC	FI	SG	KB	BL	DC	SP	DP	ZP
60	KC		*	--	--	*	--	--	*	--
	FI			*	*	--	*	*	*	--
	SG				*	*	--	--	--	*
	KB					*	--	*	*	--
	BL						*	*	*	--
	DC							--	--	*
	SP								--	*
	DP									*
120	KC		*	--	--	*	--	--	--	--
	FI			*	*	--	*	*	*	*
	SG				--	*	--	--	--	--
	KB					--	--	*	--	--
	BL						*	*	*	--
	DC							--	--	--
	SP								--	*
	DP									--
180	KC		*	--	--	*	--	--	--	--
	FI			*	--	--	*	*	*	*
	SG				--	*	--	--	--	--
	KB					*	--	--	--	--
	BL						*	*	*	*
	DC							--	--	--
	SP								--	--
	DP									--

Table 16

Matrixes of Scheff'e multiple comparison tests between all materials at 15 minutes storage time. (Stars indicate significant difference at 1% Alpha level).

	ZI	TL	DP	SG	SP	VB	KC	DC	XR	KB	ZP	FI
FI	*	*				*						
BL	*	*				*						

Table 17

Matrixes of Scheff'e multiple comparison tests between all materials at 24 hours storage time. (Stars indicate significant difference at 1% Alpha level).

	ZI	TL	DP	SG	SP	VB	KC	DC	XR	KB	ZP	FI
BL	*	*	*	*	*		*	*	*	*	*	*
ZI						*						

	TimeLine	(6.23 ± 0.01)
light-cured	XR Ionomer	(6.48 ± 0.16)
	Zionomer	(6.72 ± 0.08)
polycarboxylates	Shofu Hy-Bond	(6.5 ± 0.17)
	Durelon	(6.3 ± 0.19)
	and Zinc phosphate	(6.18 ± 0.72)

Scheff'e multiple comparison test was also performed for other storage times, at 1 percent nominal level. Results are presented in Table 18.

Storage Time Effect

To evaluate the effect of storage times (15 minutes, 30 minutes, 45 minutes, 1 hour, 2 hours, 3 hours and 24 hours) for each materials, seven separate one-way ANOVA were performed. The ANOVA Tables are presented in the Appendix Table 55 through 67. The analysis revealed a highly significant difference for all materials except Vitrabond, where there is no significant storage time effect $p = 0.0784$.

All materials show the increase in pH values with time, as expected, up to 24 hours from mixing time. The patterns of pH increase are presented graphically in Figure 3 for light-cured, Figure 4 for self-cured and Figure 5 for control groups.

Table 18

Matrixes of Scheff'e multiple comparison tests between all materials at all storage times. (Stars indicate significant difference at 1% Alpha level).

STORAGE TIME		ZI	TL	DP	SG	SP	VB	KC	DC	XR	KB	ZP	FI
15	: FI	*	*				*						
	BL	*	*				*						
30	: FI	*	*	*	*								
	BL	*	*	*	*								
	ZP	*	*										
	XR	*											
45	: FI	*	*	*	*	*							
	BL	*	*	*	*	*							
	ZP	*	*	*	*								
	XR	*	*	*							*		
	DP						*						
60	: FI	*	*	*	*	*	*	*	*	*	*		
	BL	*	*	*	*	*		*	*		*		
	ZP	*	*	*	*	*							
	XR	*	*	*	*	*							
	DP						*				*		
120	: FI	*	*	*	*	*	*	*	*	*			
	BL	*	*	*	*	*	*	*	*	*			
	ZP	*											
	KB	*											
180	: FI	*	*	*	*	*		*	*	*			
	BL	*	*	*	*	*		*	*	*		*	
	ZI						*				*	*	
1440	: BL	*	*	*	*	*		*	*	*	*	*	*
	ZI						*						

Figure 3

Acidity level as function of Storage time

for Light-cured Glass Ionomer Materials.

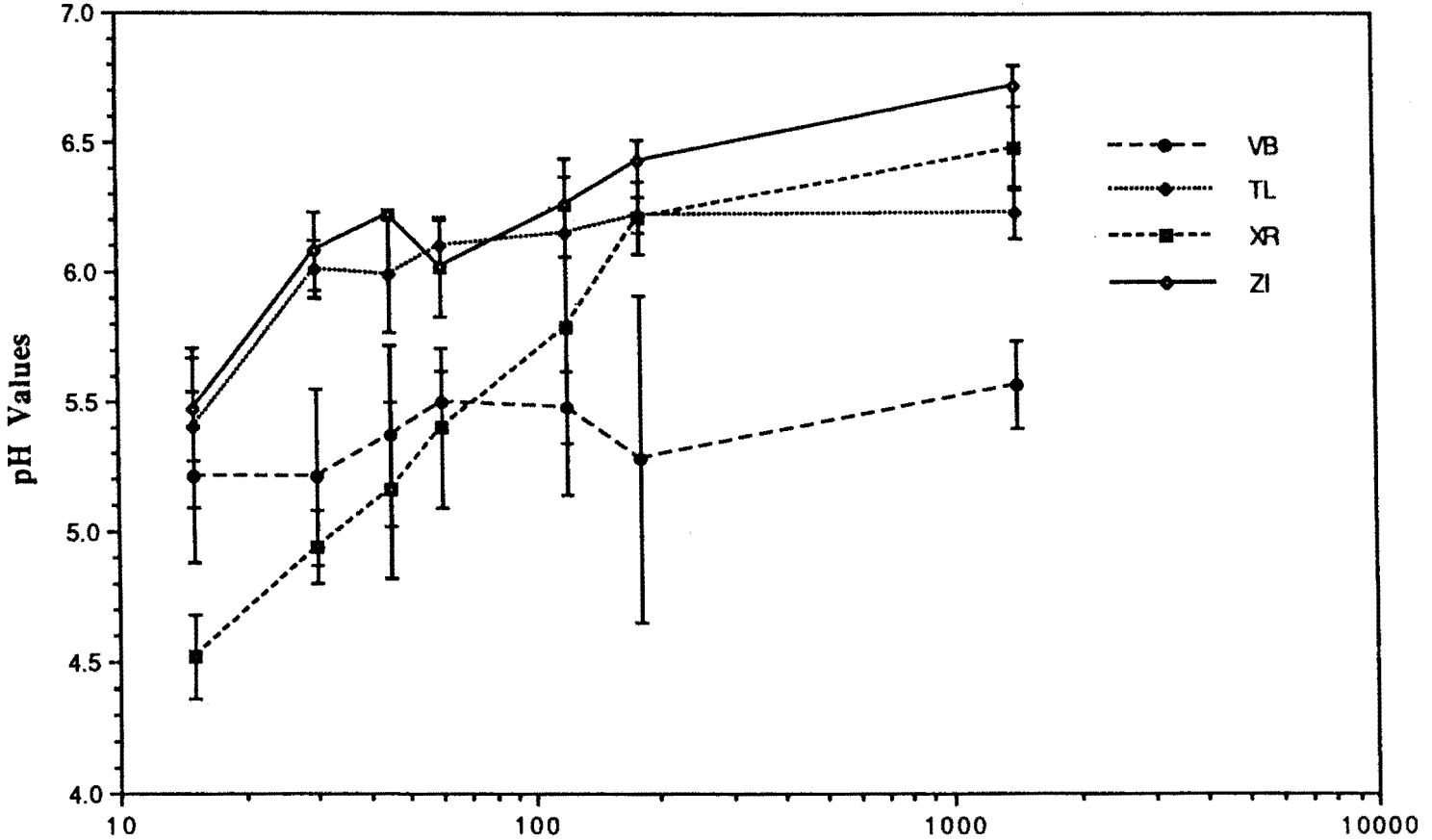


Figure 4

Acidity level as function of storage time

for Self-cured Glass Ionomer Materials.

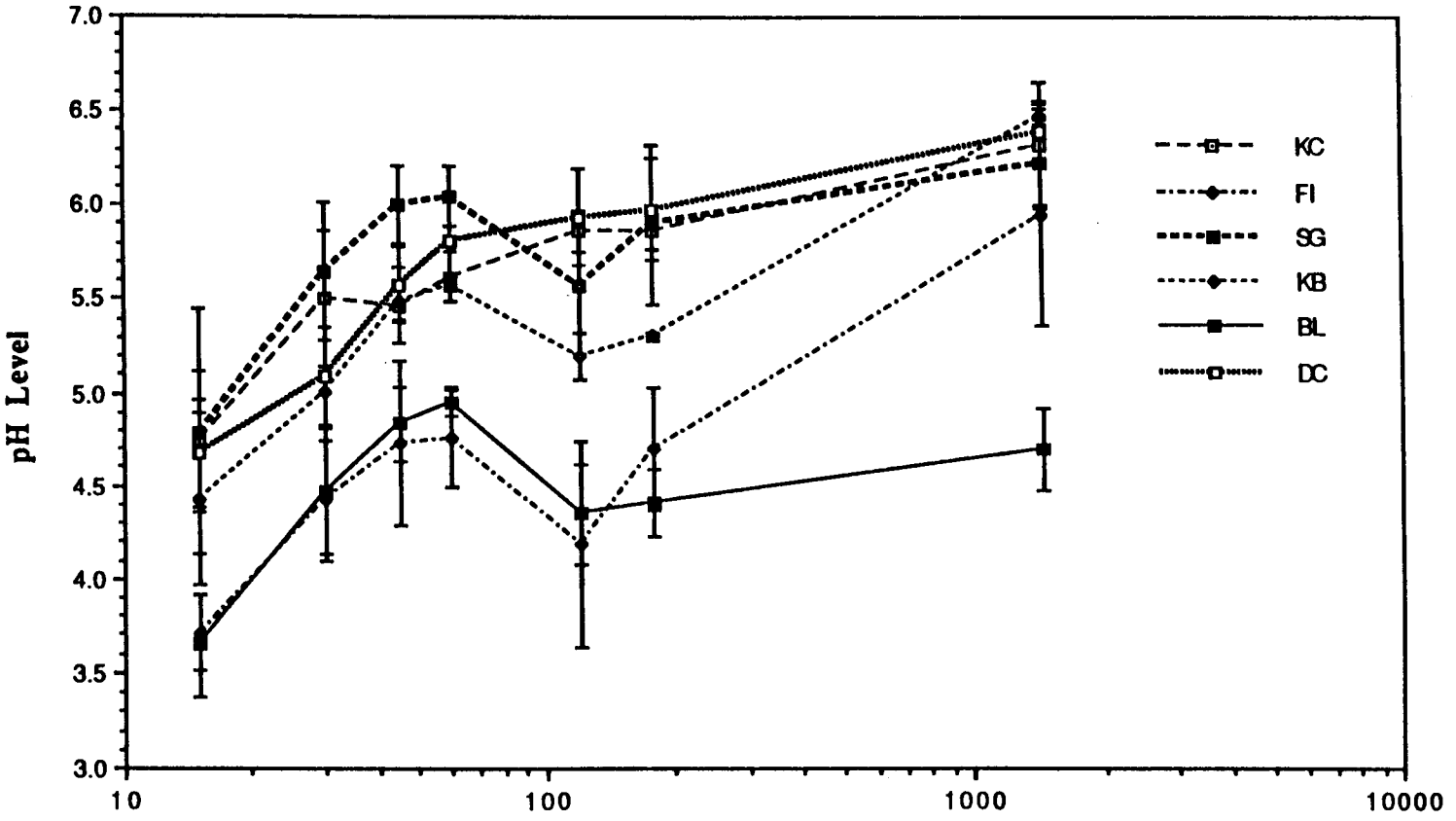
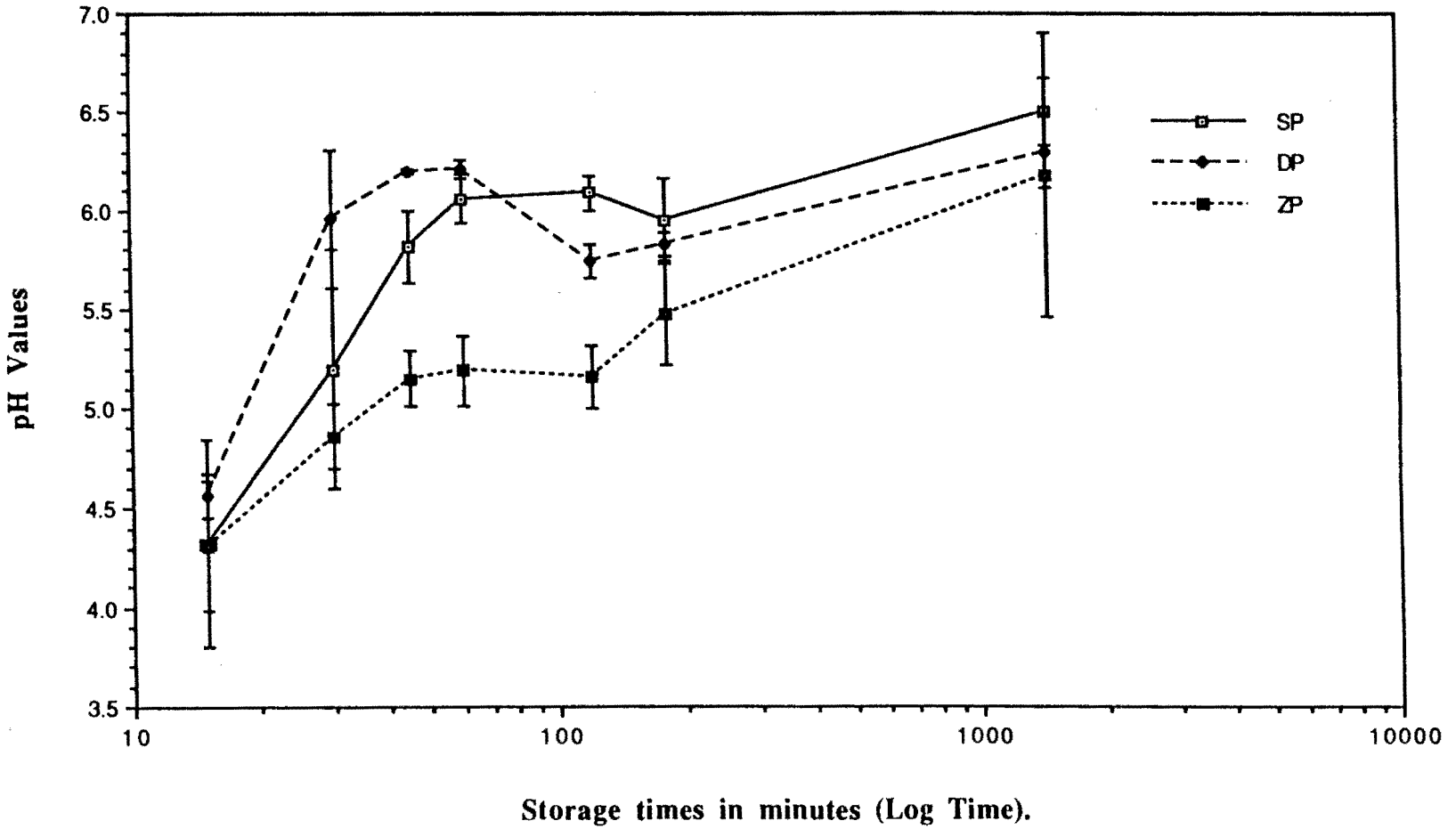


Figure 5

Acidity level of control groups as function of storage time.



DISCUSSION

The objective of this study was to evaluate in vitro the acidity of four light-cured glass ionomers, six self-cured glass ionomers, and to compare them with polycarboxylate and zinc phosphate cements. Materials were tested after seven periods of storage (15 minutes, 30 minutes, 45 minutes, 60 minutes, 120 minutes, 180 minutes and after 24 hours). Also this investigation compared the pH level of all materials. All cements tested behaved differently with respect to the pH.

Light-Cured Glass Ionomer Liners

The results show a high pH value for all light-cured materials 15 minutes after mixing with a range of 5.21 - 5.47, except for XR-Ionomer which was 4.5. This may be explained by the absence of HEMA in the XR liquid, which results in a material with an initial set that is clinically workable, but much softer than the other liners. [12]

Although the initial setting of the light-cured ionomers is fast since an initial covalent bond is formed between the methyacrylate groups on the polyacid chain, there still exists auto curing ionic bonds between the glass to the polyacrylic acid matrix. [12] This setting reaction is reflected in pH changes up to 24 hours.

Vitrabond behaved differently in that the pH did not change with time from the initial set up to 24 hours. This may reduce the chance of having free acid available to cause a pulpal reaction, however, the pH of this material after 24 hours

is still acidic (pH 5.6 ± 0.17).

Initially XR-Ionomer showed a hydrophilic behavior. After light-curing the material contracted away from the ring. When two drops of water were added, the material expanded. This supports the claims made by the manufacturer that XR-Ionomer shrinks 3% during light curing and after setting it absorbs a slight amount of water from the oral environment, resulting in a 4.5% expansion. [12] This material behavior may dehydrate the dentin and conceivably elicit pain and result in aspiration of odontoblasts into the tubules although its a light-cured material[30].

Zionomer showed the highest pH of all light-cured materials at 15 minutes (5.5 ± 0.02) and after 24 hours (6.7 ± 0.08).

Self-Cured Glass Ionomers

The data indicated a slower but similar increase in the pH for the self-cured materials from 15 minutes up to 24 hours except for Fuji Ionomer and BaseLine.

Fuji Ionomer at 15 minutes started at low pH (3.7 ± 0.20) which gave an indication that this material at early stage (0 minutes) exhibits a very low pH. This low pH may be explained since this material is used as a luting cement rather than a base/lining cements. Smith, et. al suggested that with lining cements the period of pH 2 or 3 is shorter than the luting cement for the first two minutes. Thus pulpal response is less likely with lining materials. [40]

BaseLine when compared with the other materials that are used as a base/lining cements, shows the lowest pH (3.7 ± 0.27) at 15 minutes and it shows the lowest, slowest rate reaction up to 24 hours (pH = 4.7 ± 0.22). This may indicate a less complete setting of the material.

The differences in the delivery system between BaseLine and the other

materials may account for the low pH level of this material. The active polyacid in BaseLine is in powder form mixed with glass powder and the liquid is water.

Between All Materials

Previous research [37,38,40] has implied that the pH of a setting dental cement is critical to producing pathological pulpal responses. Plant and Tyas suggested that if the pH is near 2, pulpal response depends on the duration of the low pH and is enhanced by the quantity of available acid. [41] Smith and Ruse found there was a rapid rise in pH during the first 15 minutes after mixing, showing a pH of 2 for at least 5 minutes and 3 for at least 10 minutes for all glass ionomer cements. They concluded that the early acidity of the glass ionomer cements may be a major contributor to pulp sensitivity. [38] However, none of these studies specified the exposure time that is needed for a low pH to elicit a pulp response, neither did they specify the level of pH that might cause pulp pathology.

A study done by Svare and Meyer [42] showed that acids at pH 2.8 to 2.9 induced vascular thrombosis in the pulps of rats. They conclude that if the pH is not below 2 or 3 there will be no effect on the pulp, however, that approach is misleading.

The present data supports the findings of Smith and Ruse[38] that after 15 minutes all materials show a slow increase of pH up to 24 hours. The pH at 15 minutes for all materials ranges between 3.65 - 5.47 and at 24 hours the pH ranges between 4.70 -6.72. This slow increase indicates that the setting reaction is not complete and there is still free acid present. This free acid even at late stages (after 15 minutes - 24 hours) may cause mild pulp response. So we suggested that pulp irritation may occur not only at the early stages of setting but also at the

later stages as long as the reaction is not complete and free acid still exists. The severity of pulp responses may vary depending upon the setting rate of the cement.

The pH values of self-cured materials obtained in this study are slightly higher than those reported in previous studies. [38,40] A possible explanation for the higher value could be that the pH values in this study were obtained via a deionized water bridge between the electrode and the set cements, which seemed to affect the results by 1 or 2 pH units. [38] The fact that most of these materials were mixed and measured as base/lining material and are used in a thicker mixture at a higher powder/liquid ratio (Table II), except for Fuji Ionomer and Hy-Bond polycarboxylate which are used as a luting cement may account for higher pH values.

Comparing Fuji Ionomer and Hy-Bond polycarboxylate, Fuji Ionomer shows a higher acidity level and a longer setting time than Hy-Bond polycarboxylate. This supports the finding that the initial setting of the glass ionomer cements are slow since first calcium and then aluminum ions are leached from the glass on reaction with the aqueous polyacid. [19]

Analysis of these results showed light-cured glass ionomer liners were significantly less acidic than polycarboxylates (Shofu Hy-Bond and Durelon) and zinc phosphate cement. Since the pH and the free acidity depend on the setting rate of the cement, light-cured materials will have more complete setting in the early stages than the self-cured materials. Thus, it seems unlikely that an initial pulp response would be expected with these materials.

The pH level of self-cured glass ionomer cements were similar to that of

polycarboxylate and zinc phosphate cements. This finding indicates that glass ionomer cements may be safely used in dentistry as well as the widely used polycarboxylate and zinc phosphate, as far as acidity is concerned.

All pH measurements were made at room temperature which affects the setting rate, the pH may rise more rapidly in the mouth than at the room temperature. [38]

Previous research [43,44] suggested that premature moisture contamination of the glass ionomer before completion of its setting reaction may allow fluids to contact cut dentin surfaces, thereby giving rise to sensitivity. With light-cured glass ionomer this may not occur since the setting reaction of this material will be triggered by visible light which shortens the setting time.

An unexpected sharp drop in pH was observed after 120 minutes storage time. This drop in pH was generally observed with the self-cured glass ionomers (Figure 3) and polycarboxylate (Durelon, Figure 5). This phenomena may be explained by one of the following:

1. Technical error: the drop in pH values of the materials may be caused by temperature change when the sample is removed from the humidity chamber to the room temperature (37°C - 22°C) respectively.

2. Chemical Reaction Change: the sharp drop phenomena might result from a change in the nature of the chemical reaction at that time. This assumption is more favorable than the technical error, because this drop occurs only with self-cured materials and not with light-cured materials, even though all materials were stored in the same manner. This hypothetical explanation can only be confirmed with more investigation using specific

analysis like Fourier Transform Infrared spectroscopy (FTIR).

If the initial acidity is one factor of pulp sensitivity, light-cured glass ionomer liners may reduce this factor. However, routine use of calcium hydroxide continues to be suggested[34,37] especially in deep preparations near the pulp. Proper isolation and material mainpulation remain critical to this success.

CONCLUSION

In conclusion, inflammation and possible irreversible damage to dental pulp due to prolonged exposure to acidity, should always affect the dentist's decision in choosing a particular dental material. Other factors which may be the cause of pulp sensitivity observed with glass ionomer cements must include: mechanical irritation, microleakage, bacterial contamination during cavity preparation, the preexisting condition of the tooth before tooth preparation, the depth and extent of preparation and age of the patient.

Under the conditions of this study:

1. The acidity of light-cured glass ionomer liners with exception of XR is less than that of self-cured glass ionomers, polycarboxylate and zinc phosphate cements up to 1 hour storage time.
2. Of the materials tested, BaseLine and Fuji Ionomer are the most acidic up to 24 hours. However, the pH of Fuji Ionomer rises above BaseLine at 24 hours (pH = 6 ± 0.59)
3. Almost all materials after 24 hours storage time, show an increase in pH values to a final pH approaching 7 except BaseLine which showed the lowest pH value (4.7 ± 0.22) at 24 hours.
4. From the acidity point of view, self-cured glass ionomer cements as well as polycarboxylate and zinc phosphate cements may be safely used in restorative dentistry as bases and liners.

5. In light of the results obtained in this study, light-cured glass ionomer cements appear to be a material that can be successfully used in restorative procedures as Base/Lining materials, however, further data for pulp sensitivity and clinical studies are needed.

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APPENDIX

Table 19

Two-way analysis of variance for materials and storage times.

Anova table for a 2-factor repeated measures Anova.

Source:	df:	Sum of Squares:	Mean Square:	F-test:	P value:
MATE (A)	12	100.241	8.353	39.119	.0001
subjects w. groups	52	11.104	.214		
Repeated Measure (B)	6	87.301	14.55	235.692	.0001
AB	72	26.757	.372	6.02	.0001
B x subjects w. groups	312	19.261	.062		

1

There were no missing cells found.

Table 20

One-way analysis of variance for Light-cured materials tested after 15 minutes storage time.

One Factor ANOVA-Repeated Measures for X_1 ... X_4

Source:	df:	Sum of Squares:	Mean Square:	F-test:	P value:
Between subjects	4	.266	.067	.275	.8896
Within subjects	15	3.631	.242		
treatments	3	2.817	.939	13.841	.0003
residual	12	.814	.068		
Total	19	3.897			

Reliability Estimates for- All treatments: -2.637 Single Treatment: -.221

1

Table 21

One-way analysis of variance for Light-cured materials tested after 30 minutes storage time.

One Factor ANOVA-Repeated Measures for X_1 ... X_4

Source:	df:	Sum of Squares:	Mean Square:	F-test:	P value:
Between subjects	4	.423	.106	.308	.8679
Within subjects	15	5.149	.343		
treatments	3	4.889	1.63	75.105	.0001
residual	12	.26	.022		
Total	19	5.572			

Reliability Estimates for- All treatments: -2.244 Single Treatment: -.209

1

Table 22

One-way analysis of variance for Light-cured materials tested after 45 minutes storage time.

One Factor ANOVA-Repeated Measures for X_1 ... X_4

Source:	df:	Sum of Squares:	Mean Square:	F-test:	P value:
Between subjects	4	.282	.071	.228	.9185
Within subjects	15	4.641	.309		
treatments	3	3.78	1.26	17.568	.0001
residual	12	.861	.072		
Total	19	4.923			

Reliability Estimates for- All treatments: -3.389 Single Treatment: -.239

1

Table 23

One-way analysis of variance for Light-cured materials tested after 60 minutes storage time.

One Factor ANOVA-Repeated Measures for X_1 ... X_4

Source:	df:	Sum of Squares:	Mean Square:	F-test:	P value:
Between subjects	4	.123	.031	.191	.9391
Within subjects	15	2.405	.16		
treatments	3	1.89	.63	14.67	.0003
residual	12	.515	.043		
Total	19	2.528			

Reliability Estimates for- All treatments: -4.224 Single Treatment: -.253

1

Table 24

One-way analysis of variance for Light-cured materials tested after 120 minutes storage time.

One Factor ANOVA-Repeated Measures for X_1 ... X_4

Source:	df:	Sum of Squares:	Mean Square:	F-test:	P value:
Between subjects	4	.409	.102	.46	.764
Within subjects	15	3.335	.222		
treatments	3	1.884	.628	5.196	.0157
residual	12	1.451	.121		
Total	19	3.743			

Reliability Estimates for- All treatments: -1.175 Single Treatment: -.156

1

Table 25

One-way analysis of variance for Light-cured materials tested after 180 minutes storage time.

One Factor ANOVA-Repeated Measures for X_1 ... X_4

Source:	df:	Sum of Squares:	Mean Square:	F-test:	P value:
Between subjects	4	.252	.063	.175	.9478
Within subjects	15	5.404	.36		
treatments	3	3.92	1.307	10.567	.0011
residual	12	1.484	.124		
Total	19	5.656			

Reliability Estimates for- All treatments: -4.713 Single Treatment: -.26

1

Table 26

One-way analysis of variance for Light-cured materials tested after 24 hours storage time.

One Factor ANOVA-Repeated Measures for X_1 ... X_4

Source:	df:	Sum of Squares:	Mean Square:	F-test:	P value:
Between subjects	4	.023	.006	.022	.999
Within subjects	15	3.981	.265		
treatments	3	3.716	1.239	56.106	.0001
residual	12	.265	.022		
Total	19	4.004			

Reliability Estimates for- All treatments: -45.15 Single Treatment: -.324

1

Table 27

One-way analysis of variance for Light-cured materials including control groups tested after 15 minutes storage time.

One Factor ANOVA-Repeated Measures for X_1 ... X_7

Source:	df:	Sum of Squares:	Mean Square:	F-test:	P value:
Between subjects	4	1.056	.264	.839	.5116
Within subjects	30	9.44	.315		
treatments	6	7.866	1.311	19.987	.0001
residual	24	1.574	.066		
Total	34	10.495			

Reliability Estimates for- All treatments: -.192 Single Treatment: -.024

1

Table 28

One-way analysis of variance for Light-cured materials including control groups tested after 30 minutes storage time.

One Factor ANOVA-Repeated Measures for X_1 ... X_7

Source:	df:	Sum of Squares:	Mean Square:	F-test:	P value:
Between subjects	4	1.184	.296	.888	.4831
Within subjects	30	10.005	.333		
treatments	6	8.466	1.411	22.011	.0001
residual	24	1.539	.064		
Total	34	11.189			

Reliability Estimates for- All treatments: -.126 Single Treatment: -.016

1

Table 29

One-way analysis of variance for Light-cured materials including control groups tested after 45 minutes storage time.

One Factor ANOVA-Repeated Measures for X_1 ... X_7

Source:	df:	Sum of Squares:	Mean Square:	F-test:	P value:
Between subjects	4	.239	.06	.231	.9188
Within subjects	30	7.751	.258		
treatments	6	6.626	1.104	23.551	.0001
residual	24	1.125	.047		
Total	34	7.99			

Reliability Estimates for- All treatments: -3.33 Single Treatment: -.123

1

Table 30

One-way analysis of variance for Light-cured materials including control groups tested after 60 minutes storage time.

One Factor ANOVA-Repeated Measures for X_1 ... X_7

Source:	df:	Sum of Squares:	Mean Square:	F-test:	P value:
Between subjects	4	.105	.026	.138	.9668
Within subjects	30	5.7	.19		
treatments	6	4.962	.827	26.886	.0001
residual	24	.738	.031		
Total	34	5.805			

Reliability Estimates for- All treatments: -6.241 Single Treatment: -.14

1

Table 31

One-way analysis of variance for Light-cured materials including control groups tested after 120 minutes storage time.

One Factor ANOVA-Repeated Measures for X_1 ... X_7

Source:	df:	Sum of Squares:	Mean Square:	F-test:	P value:
Between subjects	4	.29	.072	.341	.8481
Within subjects	30	6.367	.212		
treatments	6	4.642	.774	10.763	.0001
residual	24	1.725	.072		
Total	34	6.656			

Reliability Estimates for- All treatments: -1.932 Single Treatment: -.104

1

Table 32

One-way analysis of variance for Light-cured materials including control groups tested after 180 minutes storage time.

One Factor ANOVA-Repeated Measures for X_1 ... X_7

Source:	df:	Sum of Squares:	Mean Square:	F-test:	P value:
Between subjects	4	.172	.043	.178	.948
Within subjects	30	7.24	.241		
treatments	6	5.209	.868	10.259	.0001
residual	24	2.031	.085		
Total	34	7.412			

Reliability Estimates for- All treatments: -4.617 Single Treatment: -.133

1

Table 33

One-way analysis of variance for Light-cured materials including control groups tested after 24 hours storage time.

One Factor ANOVA-Repeated Measures for X_1 ... X_7

Source:	df:	Sum of Squares:	Mean Square:	F-test:	P value:
Between subjects	4	.297	.074	.349	.8423
Within subjects	30	6.371	.212		
treatments	6	4.026	.671	6.869	.0002
residual	24	2.345	.098		
Total	34	6.668			

Reliability Estimates for- All treatments: -1.862 Single Treatment: -.102

1

Table 34

One-way analysis of variance for Self-cured materials tested after 15 minutes storage time.

One Factor ANOVA-Repeated Measures for X_1 ... X_6

Source:	df:	Sum of Squares:	Mean Square:	F-test:	P value:
Between subjects	4	.438	.11	.265	.8976
Within subjects	25	10.339	.414		
treatments	5	6.786	1.357	7.64	.0004
residual	20	3.553	.178		
Total	29	10.778			

Reliability Estimates for- All treatments: -2.774 Single Treatment: -.14

1

Table 35

One-way analysis of variance for Self-cured materials tested after 30 minutes storage time.

One Factor ANOVA-Repeated Measures for X_1 ... X_6

Source:	df:	Sum of Squares:	Mean Square:	F-test:	P value:
Between subjects	4	.576	.144	.394	.8113
Within subjects	25	9.155	.366		
treatments	5	6.451	1.29	9.544	.0001
residual	20	2.704	.135		
Total	29	9.732			

Reliability Estimates for- All treatments: -1.541 Single Treatment: -.112

1

Table 36

One-way analysis of variance for Self-cured materials tested after 45 minutes storage time.

One Factor ANOVA-Repeated Measures for X_1 ... X_6

Source:	df:	Sum of Squares:	Mean Square:	F-test:	P value:
Between subjects	4	.108	.027	.095	.9831
Within subjects	25	7.077	.283		
treatments	5	5.682	1.136	16.301	.0001
residual	20	1.394	.07		
Total	29	7.184			

Reliability Estimates for- All treatments: -9.51 Single Treatment: -.178

1

Table 37

One-way analysis of variance for Self-cured materials tested after 60 minutes storage time.

One Factor ANOVA-Repeated Measures for X_1 ... X_6

Source:	df:	Sum of Squares:	Mean Square:	F-test:	P value:
Between subjects	4	.257	.064	.237	.9149
Within subjects	25	6.781	.271		
treatments	5	6.307	1.261	53.155	.0001
residual	20	.475	.024		
Total	29	7.038			

Reliability Estimates for- All treatments: -3.224 Single Treatment: -.146

1

Table 38

One-way analysis of variance for Self-cured materials tested after 120 minutes storage time.

One Factor ANOVA-Repeated Measures for X_1 ... X_6

Source:	df:	Sum of Squares:	Mean Square:	F-test:	P value:
Between subjects	4	.705	.176	.272	.893
Within subjects	25	16.185	.647		
treatments	5	14.404	2.881	32.351	.0001
residual	20	1.781	.089		
Total	29	16.89			

Reliability Estimates for- All treatments: -2.672 Single Treatment: -.138

1

Table 39

One-way analysis of variance for Self-cured materials tested after 180 minutes storage time.

One Factor ANOVA-Repeated Measures for X_1 ... X_6

Source:	df:	Sum of Squares:	Mean Square:	F-test:	P value:
Between subjects	4	.297	.074	.147	.9626
Within subjects	25	12.612	.504		
treatments	5	11.304	2.261	34.576	.0001
residual	20	1.308	.065		
Total	29	12.908			

Reliability Estimates for- All treatments: -5.801 Single Treatment: -.166

1

Table 40

One-way analysis of variance for Self-cured materials tested after 24 hours storage time.

One Factor ANOVA-Repeated Measures for X_1 ... X_6

Source:	df:	Sum of Squares:	Mean Square:	F-test:	P value:
Between subjects	4	.553	.138	.268	.8955
Within subjects	25	12.887	.515		
treatments	5	11.089	2.218	24.678	.0001
residual	20	1.797	.09		
Total	29	13.44			

Reliability Estimates for- All treatments: -2.727 Single Treatment: -.139

1

Table 41

One-way analysis of variance for Self-cured materials including control groups tested after 15 minutes storage time.

One Factor ANOVA-Repeated Measures for X_1 ... X_9

Source:	df:	Sum of Squares:	Mean Square:	F-test:	P value:
Between subjects	4	1.129	.282	.984	.4272
Within subjects	40	11.471	.287		
treatments	8	7.059	.882	6.4	.0001
residual	32	4.412	.138		
Total	44	12.6			

Reliability Estimates for- All treatments: -.016 Single Treatment: -.002

1

Table 42

One-way analysis of variance for Self-cured materials including control groups tested after 30 minutes storage time.

One Factor ANOVA-Repeated Measures for X_1 ... X_9

Source:	df:	Sum of Squares:	Mean Square:	F-test:	P value:
Between subjects	4	1.325	.331	.906	.4698
Within subjects	40	14.629	.366		
treatments	8	10.635	1.329	10.649	.0001
residual	32	3.995	.125		
Total	44	15.954			

Reliability Estimates for- All treatments: -.104 Single Treatment: -.011

1

Table 43

One-way analysis of variance for Self-cured materials including control groups tested after 45 minutes storage time.

One Factor ANOVA-Repeated Measures for X_1 ... X_9

Source:	df:	Sum of Squares:	Mean Square:	F-test:	P value:
Between subjects	4	.207	.052	.181	.9471
Within subjects	40	11.457	.286		
treatments	8	9.941	1.243	26.223	.0001
residual	32	1.516	.047		
Total	44	11.664			

Reliability Estimates for- All treatments: -4.537 Single Treatment: -.1

1

Table 44

One-way analysis of variance for Self-cured materials including control groups tested after 60 minutes storage time.

One Factor ANOVA-Repeated Measures for X_1 ... X_9

Source:	df:	Sum of Squares:	Mean Square:	F-test:	P value:
Between subjects	4	.233	.058	.206	.9336
Within subjects	40	11.316	.283		
treatments	8	10.613	1.327	60.344	.0001
residual	32	.703	.022		
Total	44	11.549			

Reliability Estimates for- All treatments: -3.854 Single Treatment: -.097

1

Table 45

One-way analysis of variance for Self-cured materials including control groups tested after 120 minutes storage time.

One Factor ANOVA-Repeated Measures for X_1 ... X_9

Source:	df:	Sum of Squares:	Mean Square:	F-test:	P value:
Between subjects	4	.357	.089	.169	.9528
Within subjects	40	21.11	.528		
treatments	8	18.826	2.353	32.97	.0001
residual	32	2.284	.071		
Total	44	21.467			

Reliability Estimates for- All treatments: -4.91 Single Treatment: -.102

1

Table 46

One-way analysis of variance for Self-cured materials including control groups tested after 180 minutes storage time.

One Factor ANOVA-Repeated Measures for X_1 ... X_9

Source:	df:	Sum of Squares:	Mean Square:	F-test:	P value:
Between subjects	4	.318	.079	.21	.9316
Within subjects	40	15.165	.379		
treatments	8	13.412	1.676	30.596	.0001
residual	32	1.753	.055		
Total	44	15.483			

Reliability Estimates for- All treatments: -3.772 Single Treatment: -.096

1

Table 47

One-way analysis of variance for Self-cured materials including control groups tested after 24 hours storage time.

One Factor ANOVA-Repeated Measures for X_1 ... X_9

Source:	df:	Sum of Squares:	Mean Square:	F-test:	P value:
Between subjects	4	.599	.15	.365	.8324
Within subjects	40	16.44	.411		
treatments	8	12.335	1.542	12.02	.0001
residual	32	4.105	.128		
Total	44	17.039			

Reliability Estimates for- All treatments: -1.743 Single Treatment: -.076

1

Table 48

One-way analysis of variance for all materials tested after 15 minutes storage time.

One Factor ANOVA-Repeated Measures for X_1 ... X_{13}

Source:	df:	Sum of Squares:	Mean Square:	F-test:	P value:
Between subjects	4	1.261	.315	.788	.5372
Within subjects	60	23.985	.4		
treatments	12	18.625	1.552	13.899	.0001
residual	48	5.36	.112		
Total	64	25.246			

Reliability Estimates for- All treatments: -.268 Single Treatment: -.017

1

Table 49

One-way analysis of variance for all materials tested after 30 minutes storage time.

One Factor ANOVA-Repeated Measures for X_1 ... X_{13}

Source:	df:	Sum of Squares:	Mean Square:	F-test:	P value:
Between subjects	4	1.44	.36	.954	.4395
Within subjects	60	22.641	.377		
treatments	12	18.077	1.506	15.845	.0001
residual	48	4.564	.095		
Total	64	24.081			

Reliability Estimates for- All treatments: -.048 Single Treatment: -.004

1

Table 50

One-way analysis of variance for all materials tested after 45 minutes storage time.

One Factor ANOVA-Repeated Measures for X_1 ... X_{13}

Source:	df:	Sum of Squares:	Mean Square:	F-test:	P value:
Between subjects	4	.28	.07	.248	.91
Within subjects	60	16.956	.283		
treatments	12	14.369	1.197	22.225	.0001
residual	48	2.586	.054		
Total	64	17.235			

Reliability Estimates for- All treatments: -3.039 Single Treatment: -.061

1

Table 51

One-way analysis of variance for all materials tested after 60 minutes storage time.

One Factor ANOVA-Repeated Measures for X_1 ... X_{13}

Source:	df:	Sum of Squares:	Mean Square:	F-test:	P value:
Between subjects	4	.079	.02	.082	.9877
Within subjects	60	14.429	.24		
treatments	12	12.933	1.078	34.582	.0001
residual	48	1.496	.031		
Total	64	14.508			

Reliability Estimates for- All treatments: -11.22 Single Treatment: -.076

1

Table 52

One-way analysis of variance for all materials tested after 120 minutes storage time.

One Factor ANOVA-Repeated Measures for X_1 ... X_{13}

Source:	df:	Sum of Squares:	Mean Square:	F-test:	P value:
Between subjects	4	.156	.039	.079	.9884
Within subjects	60	29.614	.494		
treatments	12	25.269	2.106	23.265	.0001
residual	48	4.345	.091		
Total	64	29.77			

Reliability Estimates for- All treatments: -11.65 Single Treatment: -.076

1

Table 53

One-way analysis of variance for all materials tested after 180 minutes storage time.

One Factor ANOVA-Repeated Measures for X_1 ... X_{13}

Source:	df:	Sum of Squares:	Mean Square:	F-test:	P value:
Between subjects	4	.349	.087	.211	.9316
Within subjects	60	24.887	.415		
treatments	12	21.429	1.786	24.789	.0001
residual	48	3.458	.072		
Total	64	25.236			

Reliability Estimates for- All treatments: -3.75 Single Treatment: -.065

1

Table 54

One-way analysis of variance for all materials tested after 24 hours storage time.

One Factor ANOVA-Repeated Measures for X_1 ... X_{13}

Source:	df:	Sum of Squares:	Mean Square:	F-test:	P value:
Between subjects	4	.431	.108	.31	.8702
Within subjects	60	20.856	.348		
treatments	12	16.295	1.358	14.29	.0001
residual	48	4.561	.095		
Total	64	21.287			

Reliability Estimates for- All treatments: -2.226 Single Treatment: -.056

1

Table 55

One-way analysis of variance for Light-cured Vitra bond material tested after seven different lengths of storage times.

One Factor ANOVA-Repeated Measures for X_1 ... X_7

Source:	df:	Sum of Squares:	Mean Square:	F-test:	P value:
Between subjects	4	2.105	.526	8.833	.0001
Within subjects	30	1.787	.06		
treatments	6	.634	.106	2.2	.0784
residual	24	1.153	.048		
Total	34	3.892			

Reliability Estimates for- All treatments: .887 Single Treatment: .528

1

Table 56

One-way analysis of variance for Light-cured TimeLine material tested after seven different lengths of storage times.

One Factor ANOVA-Repeated Measures for X_1 ... X_7

Source:	df:	Sum of Squares:	Mean Square:	F-test:	P value:
Between subjects	4	.246	.062	.626	.6479
Within subjects	30	2.951	.098		
treatments	6	2.459	.41	19.972	.0001
residual	24	.492	.021		
Total	34	3.197			

Reliability Estimates for- All treatments: -.598 Single Treatment: -.057

1

Table 57

One-way analysis of variance for Light-cured XR-Ionomer material tested after seven different lengths of storage times.

One Factor ANOVA-Repeated Measures for X_1 ... X_7

Source:	df:	Sum of Squares:	Mean Square:	F-test:	P value:
Between subjects	4	.858	.215	.383	.8187
Within subjects	30	16.78	.559		
treatments	6	14.724	2.454	28.633	.0001
residual	24	2.057	.086		
Total	34	17.638			

Reliability Estimates for- All treatments: -1.608 Single Treatment: -.097

1

Table 58

One-way analysis of variance for Light-cured Zionomer material tested after seven different lengths of storage times.

One Factor ANOVA-Repeated Measures for X_1 ... X_7

Source:	df:	Sum of Squares:	Mean Square:	F-test:	P value:
Between subjects	4	.015	.004	.022	.999
Within subjects	30	5.06	.169		
treatments	6	4.559	.76	36.343	.0001
residual	24	.502	.021		
Total	34	5.075			

Reliability Estimates for- All treatments: -43.67 Single Treatment: -.162

1

Table 59

One-way analysis of variance for Self-cured Ketac-Cem material tested after seven different lengths of storage times.

One Factor ANOVA-Repeated Measures for X_1 ... X_7

Source:	df:	Sum of Squares:	Mean Square:	F-test:	P value:
Between subjects	4	.173	.043	.148	.9627
Within subjects	30	8.804	.293		
treatments	6	7.121	1.187	16.924	.0001
residual	24	1.683	.07		
Total	34	8.977			

Reliability Estimates for- All treatments: -5.777 Single Treatment: -.139

1

Table 60

One-way analysis of variance for Self-cured GC Fuji I material tested after seven different lengths of storage times.

One Factor ANOVA-Repeated Measures for X_1 ... X_7

Source:	df:	Sum of Squares:	Mean Square:	F-test:	P value:
Between subjects	4	2.918	.729	1.356	.2727
Within subjects	30	16.143	.538		
treatments	6	14.34	2.39	31.817	.0001
residual	24	1.803	.075		
Total	34	19.06			

Reliability Estimates for- All treatments: .262 Single Treatment: .048

1

Table 61

One-way analysis of variance for Self-cured Shofu Type I material tested after seven different lengths of storage times.

One Factor ANOVA-Repeated Measures for X_1 ... X_7

Source:	df:	Sum of Squares:	Mean Square:	F-test:	P value:
Between subjects	4	1.541	.385	1.236	.3168
Within subjects	30	9.35	.312		
treatments	6	6.834	1.139	10.862	.0001
residual	24	2.517	.105		
Total	34	10.891			

Reliability Estimates for- All treatments: .191 Single Treatment: .033

1

Table 62

One-way analysis of variance for Self-cured Katac-Bond material tested after seven different lengths of storage times.

One Factor ANOVA-Repeated Measures for X_1 ... X_7

Source:	df:	Sum of Squares:	Mean Square:	F-test:	P value:
Between subjects	4	.51	.128	.29	.8821
Within subjects	30	13.191	.44		
treatments	6	11.664	1.944	30.555	.0001
residual	24	1.527	.064		
Total	34	13.701			

Reliability Estimates for- All treatments: -2.447 Single Treatment: -.113

1

Table 63

One-way analysis of variance for Self-cured BaseLine material tested after seven different lengths of storage times.

One Factor ANOVA-Repeated Measures for X_1 ... X_7

Source:	df:	Sum of Squares:	Mean Square:	F-test:	P value:
Between subjects	4	.498	.125	.568	.688
Within subjects	30	6.585	.22		
treatments	6	5.516	.919	20.632	.0001
residual	24	1.069	.045		
Total	34	7.083			

Reliability Estimates for- All treatments: -.761 Single Treatment: -.066

1

Table 64

One-way analysis of variance for Self-cured GC Dentin cement material tested after seven different lengths of storage times.

One Factor ANOVA-Repeated Measures for X_1 ... X_7

Source:	df:	Sum of Squares:	Mean Square:	F-test:	P value:
Between subjects	4	.71	.177	.476	.7531
Within subjects	30	11.184	.373		
treatments	6	10.186	1.698	40.829	.0001
residual	24	.998	.042		
Total	34	11.894			

Reliability Estimates for- All treatments: -1.102 Single Treatment: -.081

1

Table 65

One-way analysis of variance for Shofu Hy-Bond polycarboxylate material tested after seven different lengths of storage times.

One Factor ANOVA-Repeated Measures for X_1 ... X_7

Source:	df:	Sum of Squares:	Mean Square:	F-test:	P value:
Between subjects	4	.921	.23	.387	.8163
Within subjects	30	17.865	.596		
treatments	6	15.708	2.618	29.126	.0001
residual	24	2.157	.09		
Total	34	18.787			

Reliability Estimates for- All treatments: -1.585 Single Treatment: -.096

1

Table 66

One-way analysis of variance for Durelon material tested after seven different lengths of storage times.

One Factor ANOVA-Repeated Measures for X_1 ... X_7

Source:	df:	Sum of Squares:	Mean Square:	F-test:	P value:
Between subjects	4	.142	.035	.095	.9832
Within subjects	30	11.145	.371		
treatments	6	10.556	1.759	71.77	.0001
residual	24	.588	.025		
Total	34	11.286			

Reliability Estimates for- All treatments: -9.496 Single Treatment: -.148

1

Table 67

One-way analysis of variance for Zinc phosphate material tested after seven different lengths of storage times.

One Factor ANOVA-Repeated Measures for X_1 ... X_7

Source:	df:	Sum of Squares:	Mean Square:	F-test:	P value:
Between subjects	4	.467	.117	.281	.8881
Within subjects	30	12.473	.416		
treatments	6	9.758	1.626	14.378	.0001
residual	24	2.715	.113		
Total	34	12.94			

Reliability Estimates for- All treatments: -2.562 Single Treatment: -.115

1

APPROVAL SHEET

The thesis/dissertation submitted by: **NAJWA M. JOWHARJI**
has been read and approved by the following committee:

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The final copies have been examined by the director of the thesis/dissertation and the signature which appears below verifies the fact that any necessary changes have been incorporated and that the thesis/dissertation is now given final approval by the Committee with reference to content and form.

The thesis/dissertation is therefore accepted in partial fulfillment of the requirements for the degree of (**Master Of Science**).

11/28/90
Date

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