VALIDITY OF LABORATORY TESTS

IN PREDICTING CLINICAL BEHAVIOR OF SILICATE CEMENT

by

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A Thesis

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TABLE OF CONTENTS

LIST OF FIGURES

Page

FIGURE

LIST OF TABLES

Page

TABLE

INTRODUCTION

The silicate cements were first introduced to dentistry in England by Fletcher in the 1870's (1, 2). Following a series of similar materials, all of which failed clinically, Ascher's Artificial Enamel (3) was placed on the American market. This was probably the first successful silicate introduced to this country, and the composition of silicate cement has changed very little since that time.

Silicate cements are supplied to the dental practitioner in the form of a powder and a liquid. The powders are essentially acid soluble glasses composed of silica (SiO_2), alumina (Al_2O_3), and lime (CaO). (4) These ingredients, together with fluxes (generally CaF_2), are fused at 1400° C. (2550° F.), crazed by rapid cooling and ground to the powder form (5). The liquids are dilute solutions of phosphoric acid (52% PO_4, 39% water) buffered by the addition of aluminum, zinc and magnesium phosphates (4).

The silicate cements should be the ideal restorative materials for anterior interproximal and gingival cavities because of the ease of cavity preparation, the ease of insertion and finishing, and their excellent esthetic properties. However, this material fails clinically presumably because of its inherent low edge strength and relatively high solubility and disintegration (6). Paffenbarger, in 1938, concluded from a thorough survey of dental practitioners that the life expectancy of a silicate restoration was three to five years (4). Uebele, reporting on research at the National Bureau of Standards, between 1937 and 1948,

stated that the average life of a silicate was three to four years (7). Coy reported in 1958, as the result of evaluating 123 silicate restorations, that only 44 had lasted through five years of service (8). Fusayama implies from a comparative study of acrylic and silicate restorations that the latter are inferior and that five years after placement a large majority are discolored and abraded (9).

Throughout these studies the solubility and disintegration, as well as the relatively low edge strength, have been implicated as the major cause of failure in the silicate restorations. In 1905 Hinkins published the concept that there are two prime causes to be considered in the failure of cement restorations: (1) abrasion, and (2) loss of substance due to the solvent action of the saliva or acids produced by bacteria (10). Henschel feels, as the result of observing thousands of silicate restorations over 20 years, that the solvent action of saliva is negligible when compared to the action of organic acids concentrated in the dental plaque. He states this after observing, in large interproximal silicates, that the labial and lingual surfaces remain relatively intact, while the majority of disintegration is evident mainly in the protected interproximal area (11).

To date no basic research has been reported on the clinical evaluation of the silicate cements and the possible correlation between laboratory tests and the clinical behavior of the silicates.

The laboratory tests that are presently used to evaluate dental materials have evolved from testing those materials which have proven clinically acceptable (4). Since clinical acceptance is a very qualitative parameter, it cannot be safely concluded that a material which passes the established laboratory requirements will give satisfactory clinical service. In this study a more quantitative clinical evaluation

technic was used.

All of our current knowledge of the effects of manipulative variables on the physical properties of silicate cements has been derived from the use of laboratory tests with no real understanding of the possible correlations to the clinical behavior of the material. It was, therefore, the purpose of this investigation to determine if the presently used laboratory tests can validly predict the clinical behavior of silicate cement.

GENERAL APPROACH

In order to test the prediction of clinical performance by laboratory tests, technics presumed to result in different clinical behaviors were selected. In this manner, changes in clinical behavior could be correlated to changes in the results of laboratory tests. The technics selected were (1) the conventional matrix-strip technic (a technic long established as being satisfactory), (2) the iron-on (or Olsen) method (presumably producing better adaptation and surface characteristics than the conventional technic but also possessing lower strength and increased solubility (12)), and (3) the conventional technic using a thin mix (presumably producing lower strength and higher solubility than the conventional technic (4)). This last technic was utilized in order to isolate the factors of strength and solubility from the placement method since in the pilot study the iron-on technic exhibited a reduced strength and an increased solubility. These three technics were placed randomly in class III cavities and evaluated by comparing surface replications taken at one week and three months. It is recognized that three months is a short span for a clinical study; however, time limitations prevented a longer period. These restorations will continue to be examined at six-month intervals subsequent to this study.

The laboratory tests of transverse strength and solubility were utilized in this study because of the implication of these properties in the clinical failure of this material. The compressive strength of

materials has been utilized almost exclusively as a measure of this property, probably because of the ease of sample manufacture and testing. However, the use of transverse strength is more applicable to the stresses imposed on a filling material under clinical conditions. Transverse strength is bending strength and is a composite of compressive, tensile and shear strengths. Bending strength is related to edge strength since an edge of a restoration in bending is subject to tensile, compressive and shear stresses (13). Because of this relationship to edge strength and the fact that a compressive sample cannot be manufactured with the iron-on method, the transverse strength test was utilized in this study.

The standard American Dental Association test for solubility utilizes distilled water as the solvent (14). However, when the solubility of silicate and zinc phosphate cements are compared, using distilled water as the solvent, silicate cement exhibits approximately three to four times the solubility of zinc phosphate cement. On the other hand, when a dilute organic acid is used as the solvent, the zinc phosphate cements have the higher solubility (15). Because of this phenomenon and the fact that silicate cements withstand the solvent action of the oral environment better than zinc phosphate cements, organic acids were employed in this study in addition to distilled water. Specifically, distilled water (ion free) plus four organic acids, lactic, acetic, citric, and EDTA (ethylene diamene tetraacetic acid) were utilized as solvents. The EDTA was employed for the purpose of evaluating a chelation theory in regard to the mechanism of the solubility of silicate cements.

METHODS AND MATERIALS

Laboratory Tests

Materials. A single manufacturer's silicate cement was used in this study (S. S. White New Filling Porcelain). The powder from one bottle each of shades one through six was placed in a 2-liter, square-sided jar having a self-sealing lid. This powder was then thoroughly mixed by rotating the jar in a lathe for two hours at 30 rpm. The powder was then stored in this jar for the duration of the study. An appropriate amount of liquid, of the same batch number (20164121), was purchased at the beginning of the study and a fresh bottle utilized each week.

Material Manipulation. The atmospheric environment of the laboratory in which all samples were manufactured was kept fairly constant with the aid of air conditioning. The temperature was maintained at 74° ± 1° F. and the relative humidity was approximately 35 to 40%.

Two powder/liquid ratios were used in this study: one for manufacturing the iron-on and conventional samples and one for producing the conventional-thin samples. The conventional ratio of .36 gm powder to .1 cc liquid (1.44 gm/.4 cc) was selected as being near the optimum for the clinical placement of both the iron-on and conventional restorations. The thinner ratio of .33 gm powder to .1 cc liquid (1.32 gm/.4 cc) was selected as being the thinnest ratio which would probably be used clinically. Final judgment on the selection of these two ratios was made by Dr. Kenneth R. Cantwell, Head, Department of Operative Dentistry, University of Oregon Dental School.

Figure 1

Instruments and Equipment Used in Sample Manufacture

The powder for the manufacture of each mix of the cement was weighed out on a Torsion balance (accuracy ± .01 gm). A check on the accuracy of this method of dispensation was made by reweighing ten portions of powder on a Mettler balance (accuracy ± .01 mg). The results of this check showed a mean weight of powder ± the standard error of the mean of 0.36 ± .005 gm. The liquid was proportioned with a B-D Yale Tuberculin Syringe (with markings to .01 cc). A check on this method of liquid proportioning was made by weighing, on a Mettler balance, ten 0.1 cc dispensations and converting to cc by dividing the weight by the density of the liquid (1.5152 gm/cc). The results of this check indicated a mean dispensation ± the standard error of the mean of 0.1 ± .0007 cc.

The mixing of the powder and liquid was accomplished on a standard 6 x 3 x 3/4 inch glass slab with a stellite spatula. The temperature of the glass slab was maintained at 74° F. The powder for each mix of cement was placed on one end of the glass slab and divided into four portions of 1/2, 1/4, 1/8 and 1/8. The liquid was placed on the slab just prior to mixing and the 1/2 portion of powder was then immediately incorporated, followed by the 1/4, 1/8, and final 1/8 portion. All powder was incorporated in the liquid by 25 seconds, and the mix was completed by 45 seconds. Care was taken during mixing to limit the area of spatulation to 1-1/4 inches in diameter. The area of the glass slab wetted by the cement during spatulation has a decided effect on the consistency of the mix, for as the area increases the cement assumes a thicker consistency.

Figure 1 shows the instruments utilized in the manipulation of the silicate cement in this study.

Sample Preparation. All samples were made in glass molds formed by cementing ground microscope slides to two-inch square glass plates. The sample size was 9 x 2.5 x 1 mm. A plastic spacer was inserted into the mold for the purpose of controlling the length, which was made consistent with the transverse strength testing apparatus. The thickness of the samples varied slightly with the thickness of the microscope slides (0.95 to 1.05 mm.). This variance, which was only important in the strength samples, was accounted for by measuring each sample prior to fracturing. The dimension of 2.5 mm. was selected because this width approximated the clinical situation and allowed for each margin of the iron-on sample to be adapted separately.

Prior to mixing the cement, the molds were lightly lubricated with a mixture of wax and benzene and placed on a constant-temperature surface (see Figure 1) in order to bring the mold temperature to 37° C. Preliminary work in the pilot study indicated that the physical properties of the iron-on samples were affected by the mold temperature. Appendix 1 contains the data from this study.

Two samples, an iron-on and a conventional, were made from each mix of cement. The conventional-thin samples were made separately. A second technician prepared the conventional samples.

The iron-on samples were produced as follows: Beginning 45 seconds after commencing to mix the powder and liquid, small increments of cement were carried to the mold cavity with a Gregg 4 & 5 plastic instrument. The cavity was slightly overfilled by one minute 30 seconds and the majority of the excess removed until all margins were clear. At this point cocoa butter was applied to the surface of the sample and the remainder of the excess cement was removed immediately with the Gregg 4 & 5

Table 1

Procedure for Manufacture of Silicate Samples
for Transverse Strength and Solubility Tests

Operation	Time	
	Iron	Conventional
Mix	to 0'45"	to 0'45"
Begin to fill mold	0'50"	0'50"
Remove excess	1'30"	1'30"
Apply cocoa butter	1'40"	—
Begin ironing	1'45"	—
Place matrix	—	1'40"
Place in 37° C., 100% rh oven	—	2'30"
Complete ironing	4'00"	—
Remove spacer	6'00"	6'00"
Remove matrix	—	6'05"
Place cocoa butter	6'10"	6'10"
Place in 37° C., 100% rh oven	6'30"	6'30"

instrument. Using this same instrument, lightly covered with cocoa butter, the surface of the sample was lightly burnished using an ironing motion and working from the center of the sample toward the margins. This ironing action was continued until the sample became quite hard and no further smoothing of the surface was possible (about four minutes after starting to mix the cement). At six minutes the plastic spacer controlling the length of the sample was removed, excess cocoa butter applied to all exposed surfaces and the mold, with the sample, placed in a 37° C., 100% relative humidity oven.

The conventional samples were produced by beginning to fill the mold at 45 seconds after commencing to mix the powder and liquid. The mold cavity was slightly overfilled by the addition of small increments, using a Gregg 4 & 5 plastic instrument. After one minute and 30 seconds, a mylar matrix strip was placed across the cement, followed by a glass plate. A two-inch C clamp was used to hold the matrix strip and glass plate in place. At two minutes and 30 seconds the mold and sample were transferred to a 37° C., 100% relative humidity oven. At six minutes the C clamp, matrix and spacer were removed, the exposed surfaces of the sample covered with cocoa butter and the samples, still in the mold, returned to the 37° C., 100% relative humidity oven.

A 36-gauge platinum wire was embedded in each solubility sample so the specimens could be suspended in the solvents.

Table 1 contains the timing sequence for the manufacture of silicate samples in this project.

Figure 2 illustrates the iron-on and conventional samples in their respective molds.

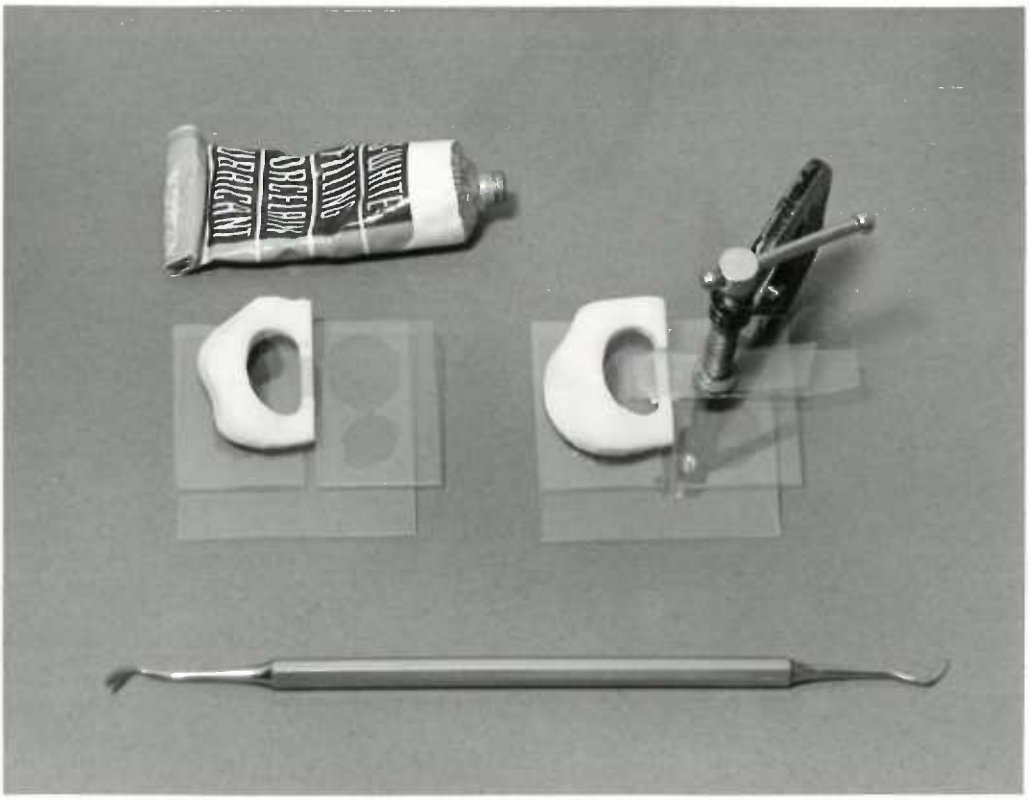


Figure 2

Iron-On and Conventional Samples in Glass Molds

Table 2

Procedure for Finishing and Testing
Silicate Samples

Operation	Time	
	Iron	Conventional
Polish surface	—	21 hrs (24 hrs for S_t)
Remove excess cocoa butter	21 hrs (24 hrs for S_t)	21 hrs (24 hrs for S_t)
Test S_t samples at this point		
For Solubility Samples:		
Place in CCl_4	21 hrs	21 hrs
Remove and blot dry	24 hrs	24 hrs
Place in tared weighing bottle	24 hrs	24 hrs
Weigh bottle and samples	24 hrs	24 hrs
Add 4 ml solvent	24 hrs	24 hrs
Remove samples	48 hrs	48 hrs
Evaporate solvent at 85° C.	48 hrs	48 hrs
Raise temp. to 110° C. (when all visible liquid evaporated)		
Record weight of bottle and residue (when weight stabilized)		

Sample Storage and Finishing. All samples were stored at 37° C. and 100% relative humidity until tested. The transverse strength specimens were stored for 24 hours, at which time the conventional samples were removed, wiped clean and the surface polished with a one-inch medium, cuttle, Moyco disk lubricated with cocoa butter. The surface flash was removed by drawing the edges of the moist samples lightly over 600 grit emery paper. The samples were then measured in width and thickness and fractured. The surfaces of the iron-on samples were not polished. If a flash was present, this was removed in the same manner as with the conventional samples. The specimens were then measured and fractured.

The solubility samples were removed from the 37° C., 100% relative humidity oven at 21 hours, wiped clean and placed in carbon tetrachloride for three hours. This procedure was necessary to remove all traces of the cocoa butter which might influence the solubility determinations. Two samples each were placed in 25 ml of fresh CCl_4 and at 24 hours the samples were removed, wiped dry, and placed in tared weighing bottles for the solubility tests.

Table 2 contains the itemized procedure for finishing and testing the silicate samples in this project.

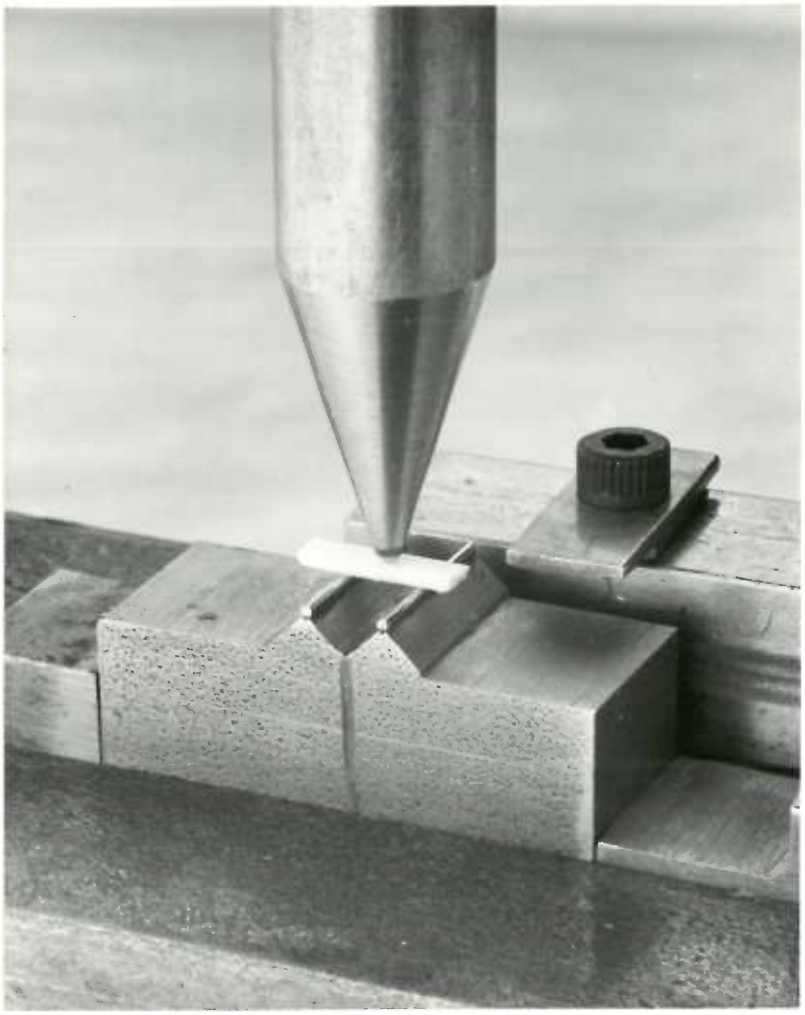


Figure 3

Specimen Platform, Positioner, and Load Applicator
for Transverse Strength Test

Testing Transverse Strength. The specimens were fractured at 24 hours in an Instron Testing machine which automatically records the load being applied to the sample at the point of failure. In this test the upper surface of the sample is subject to compressive stress while the lower surface is under tensile stress. Because tensile stresses are more critical in brittle materials such as silicate cements, the ironed and polished surfaces of the samples were placed downward. Figure 3 illustrates the testing apparatus. The samples were supported by two parallel steel rods, .030 inch diameter, which were located 5 mm. apart on the loading platform. Load was applied to the center of the sample, in the width dimension, through a 1/16-inch hardened steel ball at a rate of 0.01 in/min. Transverse strength was computed through the following formula:

$$S_t = \frac{3LP}{2bh^2}$$

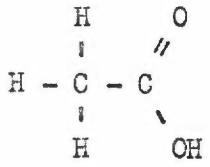
where L = distance between support rods
P = load at fracture
b = width of sample
h = thickness of sample

Testing Solubilities. Twenty-four-hour-old samples were used in this study instead of the standard one-hour sample specified by the American Dental Association Specification Test No. 9 on silicate cement. The primary rationale for this change in procedure was that the conventional samples in this investigation were subject to the same polishing technic as the clinical restorations; that is, finishing the surface with cuttlefish disks lubricated with cocoa butter when the silicate is at least 24 hours old. Also, the presence of cocoa butter on the surface of both the conventional and iron-on samples necessitated that some means be used to remove it without affecting the silicate cement, prior to testing the samples for solubility. Carbon tetrachloride

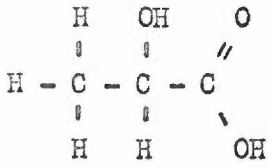
Table 3

Structural Formulas of Acids Used in this Study

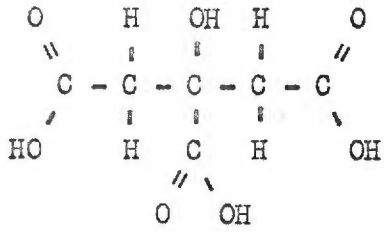
Acetic
CH_3COOH



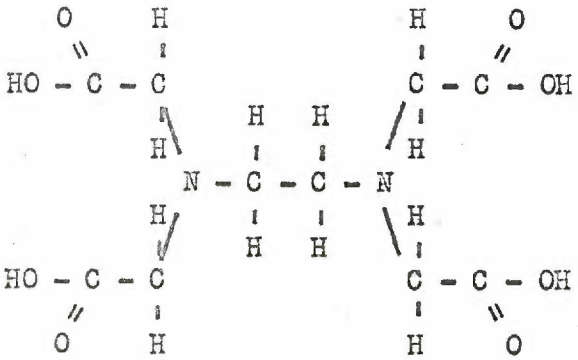
Lactic
$CH_3CHOHCOOH$



Citric
$(COOH)CH_2C(OH)(COOH)CH_2COOH$



EDTA
$(COOHCH_2)_2N(CH_2)_2N(CH_2COOH)_2$



was used for this purpose, and three hours was found to be sufficient time for complete removal of the cocoa butter. A check on the possible solubility of silicate cement in carbon tetrachloride was made. The results of this test revealed that no loss of substance had occurred after 24 hours storage in this medium.

After three hours storage in carbon tetrachloride the samples were removed, blotted dry, and two specimens each were suspended in 10 ml. tared weighing bottles and weighed. Immediately following the weighing, 4 ml. of solvent were added with a 4 ml. pipet, and the bottles stored for 24 hours at 37° C. The quantity of solvent used in this test corresponds to the same amount used in the A.D.A. test on a surface area-to-volume ratio. A check on this ratio was made by determining the solubility of an A.D.A. Specification sample (24 hours old and polished) in 50 ml. of the lactic acid solvent. The results of this test showed that the percent solubility was approximately the same for both sample sizes.

Five solvents were utilized in this investigation: distilled water (as specified in the A.D.A. test) in which the foreign ions were reduced to less than three parts per million by passing the water through an ion exchanger, and four organic acids, namely acetic, lactic, citric and EDTA. The first three acids have been used extensively in dental research (15, 16, 17), and the EDTA was employed to evaluate a chelation theory for the solubility of the silicate cements. Table 3 contains the structural formulas for the four acids. All acid solutions were produced by adding an appropriate amount of the acid to one liter of distilled water to make a 0.001 M solution. The pH was subsequently established at four by titration with 2N NaOH. Three drops of 5% thymol were added to each liter to prevent the growth of microorganisms.

Prior to computing the percent solubilities, the amount of residue remaining after evaporating 4 ml. samples of the organic acids was determined. The amount of solvent residue was then subtracted from the total residue to obtain a corrected figure for the samples. Appendix 2 contains this data as well as the data sheet utilized in this study.

After 24 hours the samples were removed from the weighing bottles and the solvent evaporated at 85° C. When all traces of liquid were gone from the bottles, the temperature was raised to 110° C. until a constant weight was achieved.

All weighings in this study were performed on a Mettler Type B6 analytical balance (accuracy 0.01 mg).

Clinical Evaluation

Materials. The same silicate cement was used in this phase of the study as was used in the laboratory tests. All teeth were restored with one of the first six shades of powder, depending on the selection made with the shade guide. A fresh bottle of liquid was used each week.

Material Manipulation. The manipulation of the cement in this phase of the project was the same as used in the laboratory. The atmospheric environment of the operatory was not controlled by air conditioning; consequently, a greater range of temperatures and humidities was experienced.

Patient Procurement and Management. All patients utilized in this study were obtained from the general clinic of the University of Oregon Dental School and consisted of teenagers with an age range of 13 to 16. Following completion of the silicate restorations, the patients were placed on a three-, six- and twelve-month recall system. Subsequent to

this period the restorations will be observed every six months.

General Operative Procedures. Following the administration of local anesthetic, the teeth to be restored were isolated with a rubber dam and cavities were prepared with a conventional belt-driven handpiece using a carbide 330 bur. The gingival and incisal retention was placed with 1/2 round steel burs. Wherever possible, cutting instruments were employed to finish the cavosurface margins. Prior to restoring the prepared teeth, an Elasticon impression (Kerr Mfg. Co.) was taken to record the shape and extent of the cavity for possible correlation to future marginal failures that might be attributed to cavity preparation. All exposed dentin was lined with calcium hydroxide (Pulpdent liquid) prior to placing the silicate cement. Care was taken to remove all traces of the liner from the enamel surfaces. If the cavities were unusually deep, a base of zinc oxide-eugenol followed by zinc phosphate cement was used. In the case of a minute pulpal exposure, Metimyd (Schering) carried on an asbestos disk was placed immediately over the exposure and covered with zinc oxide-eugenol, which in turn was covered with zinc phosphate cement.

The cavities in this study were divided into two types, depending on the access, or approach to the cavity. These types were facial and lingual. This division was necessary because of the relative differences in difficulty of restoring these two types of cavities.

Placement of Iron-On Restoration. Prior to mixing the cement a mylar matrix strip lightly lubricated with cocoa butter was placed through the contact point and positioned so that both the gingival and incisal margins were covered. The cement was placed in small increments with a Gregg 4 & 5 plastic instrument until the cavity was just slightly

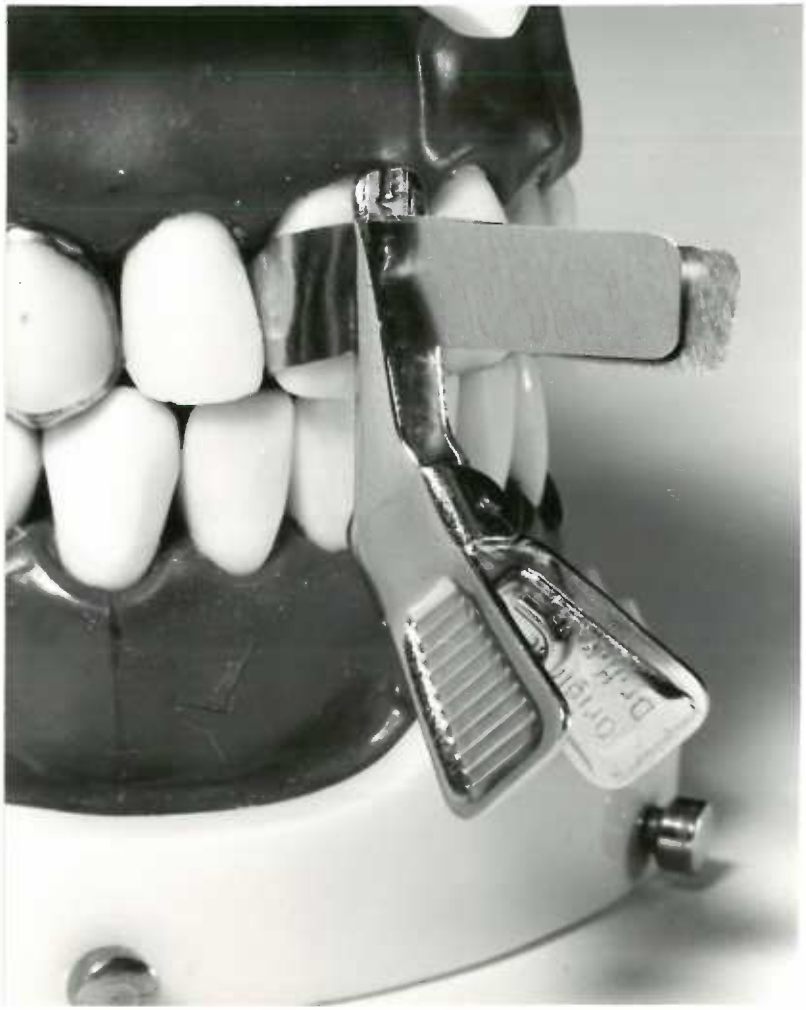


Figure 4

Premier Hawe Matrix Strip and Retainer

overfilled. At this point any gingival or gross excess was removed and the mylar strip drawn through the contact area, taking care to prevent the strip from moving incisally while it was passing over the cement. This procedure resulted in producing an initial contour to the restoration. Following this step, the excess cement was removed from the marginal areas and any gross movement of the restoration caused by drawing the mylar strip across the material was corrected. After this step, the cement was lightly covered with cocoa butter and a light ironing motion started, directing the movements of the Gregg 4 & 5 plastic instrument from the bulk of the restoration toward the margins. As the cement began to set (forming an "artificial skin") the final contour was established and the ironing procedure was continued until no further improvement of the surface could be made. For the purposes of this study, special care was given to the labial margins of facial cavities and the lingual margins of lingual cavities. This procedure was followed because it was these areas which were recorded and used for subsequent comparison. After completion, the cement was covered with cocoa butter to prevent dehydration. Fifteen minutes following completion of the last restoration, the rubber dam was removed. Prior to dismissing the patient, instructions were given to discontinue tooth brushing in the restored area for the remainder of the day.

Placement of Conventional Restoration. Before mixing the cement, a Premier Hawe 0.0035 inch gold plated aluminum matrix strip was adapted to the tooth to be restored. Figure 4 illustrates this matrix technic in use. The cavity was slightly overfilled with small increments, using a Gregg 4 & 5 plastic instrument. Immediately

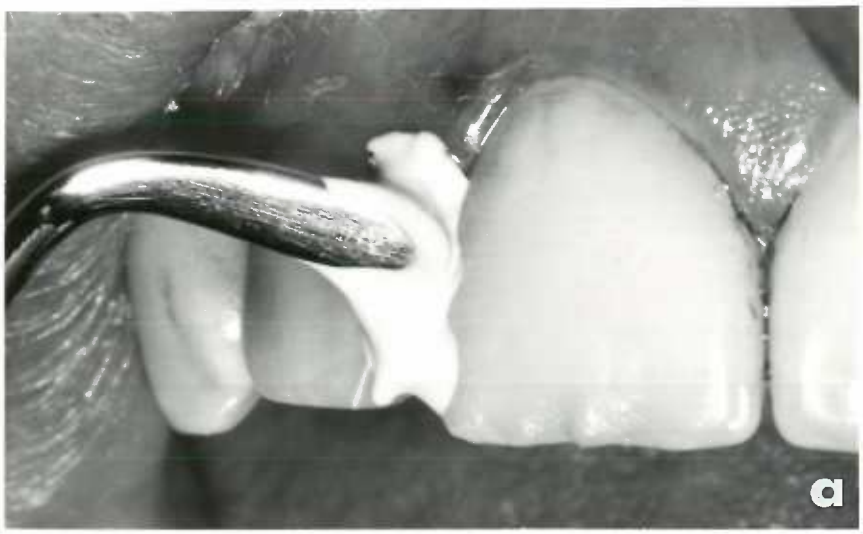
following the placement of the cement, the matrix strip was drawn across the restoration and secured in place with a Premier Striptite Retainer. The use of this retainer precludes the operator from having to hold a matrix strip while the cement is setting and removes the hazard of movement during gelation. The band was removed in four to five minutes or when an additional portion of the cement placed near the restored tooth had set. Upon removal of the band the restoration was immediately covered with cocoa butter. If a gross excess of cement was present, it was removed after fifteen minutes, taking care not to remove this excess to the margins. The rubber dam was removed fifteen minutes after placing the last restoration and the patients were instructed to discontinue tooth brushing in the restored area for the remainder of the day.

Polishing. All conventionally placed restorations were polished in one week. This procedure consisted of removing the facial excess, and lingual excess where access permitted, with slowly revolving fine cuttlefish disks lubricated with cocoa butter. The bulk of the lingual excess was removed with carborundum green stones (Phoenix) lubricated with cocoa butter. The gingival contour was established with fine and extra-fine cuttlefish strips, again well lubricated with cocoa butter.

The iron-on restorations were not polished.

Recording Restoration. In order to evaluate the influence of the placement technics on the clinical behavior of the silicate cement, some objective means of recording the surface and margins of the restorations must be used. In order to accomplish this, an impression was made (using Kerr Syringe Elasticon) of the accessible surface of the restoration at the time of polishing (seven days after insertion

for the iron-on technic) and at three months. These impressions were then silver plated in order to provide a positive replica of the restoration and surrounding tooth structure. Figure 5 illustrates this entire procedure. The silver-plated replicas were then photographed to produce a picture of about eighteen times magnification. The seven-day and three-month pictures were then compared and rated as to whether the given restoration exhibited a change or no change in surface characteristics and marginal integrity. Appendix 3 contains the silver plating and photographic procedures.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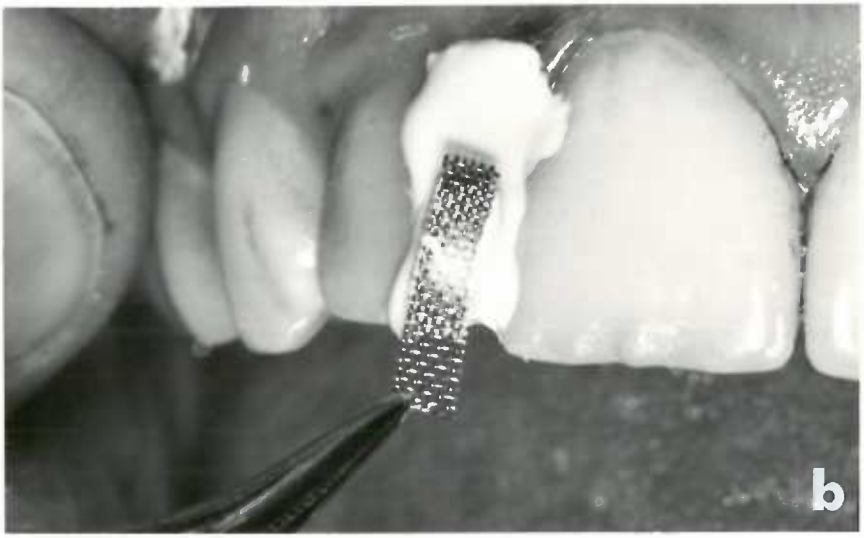
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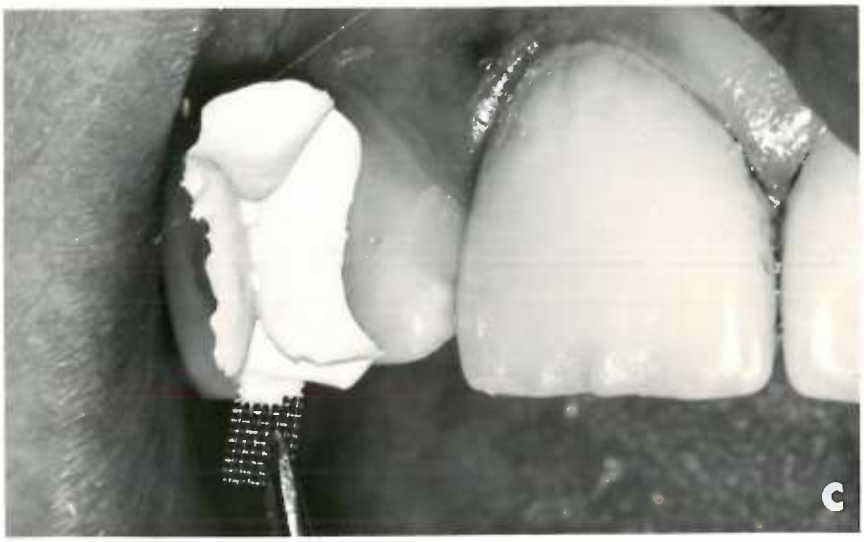
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Figure 5

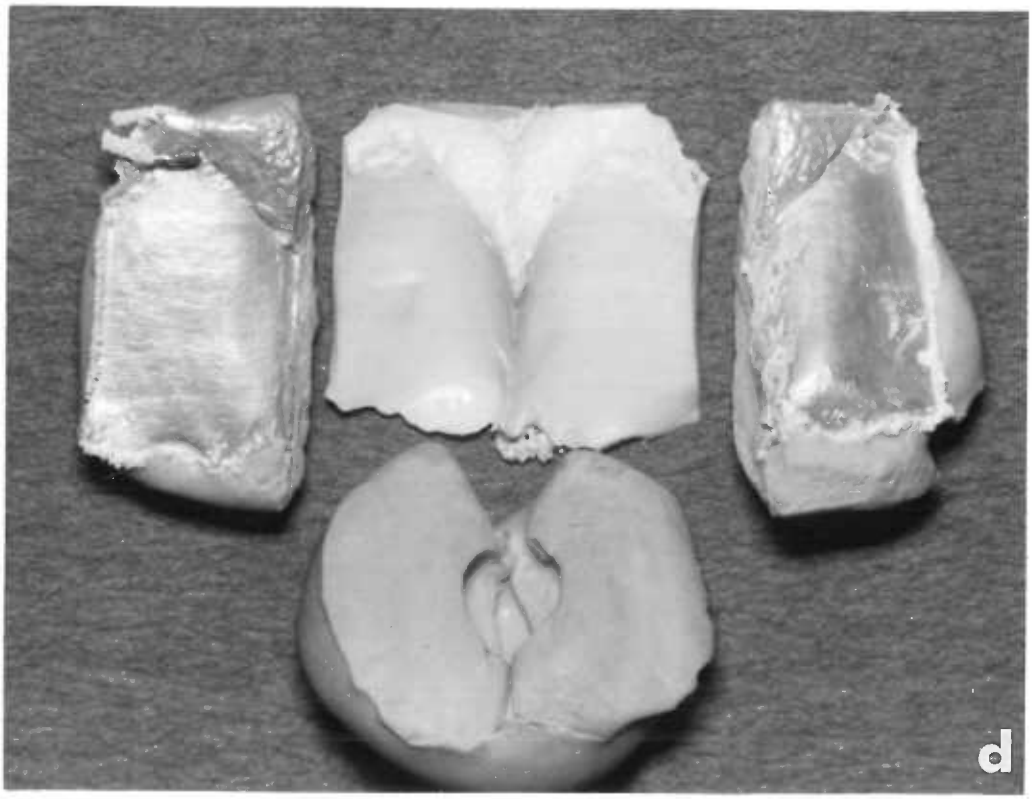
Procedure for Recording Clinical Restorations

a - Placing Impression Material

b - Placing Wire Screen

c - Impression Removed from Restoration

d - Stone Model of Cavity Preparations, Impression of Restorations and Silver-Plated Replications



EXPERIMENTAL DESIGN

The transverse strength was analyzed with respect to three placement technics. Each technic consisted of ten samples. The solubility tests were analyzed with respect to three placement technics and five solvents. Each experiment consisted of fifteen observations and was replicated eight times.

In order to minimize the variation of spatulation, an iron-on and a conventional sample were produced from the same mix of cement. A second operator was utilized to manufacture the conventional samples.

The statistical evaluation of the clinical phase of this study was made by converting the percent values of the restorations exhibiting no change in surface and marginal integrity into respective arc sines. These values were then analyzed by analysis of variance.

Table 4

Influence of Insertion Technics on Transverse Strength and Solubility of a Silicate Cement

Insertion Technic	Transverse Strength psi	% Solubility in Various Media				
		Water	Acetic	Lactic	Citric	EDTA
Iron-On	3,000 ± 150	0.47 ± .21	0.65 ± .22	0.50 ± .27	0.76 ± .20	1.35 ± .18
Convent.	3,800 ± 300	0.56 ± .12	0.80 ± .17	0.56 ± .13	0.96 ± .22	1.46 ± .18
Conv.-Thin	3,600 ± 200	0.56 ± .09	0.77 ± .10	0.60 ± .11	0.83 ± .06	1.36 ± .15

values are $\bar{X} \pm S_{\bar{X}}$ (.95)

RESULTS AND DISCUSSION

Laboratory Tests

The results of the laboratory phase of this investigation are contained in Table 4. The values for the transverse strength represent the means of ten specimens, while the values for solubility represent the means of eight separate tests containing two samples each.

Transverse Strength. The results of the transverse strength test were subject to analysis of variance, at the 95% level of significance, to test the hypothesis that the mean strengths of the three insertion technics were the same. A significant F ratio was obtained from this analysis. Orthogonal comparisons were used to test for possible differences between the iron-on and conventional technics and between the conventional and conventional-thin technics. Table 5 contains this statistical evaluation.

Table 5

Analysis of Variance with Orthogonal Comparisons for Transverse Strength

Source of Variation	Degrees of Freedom	Sum of Squares	Mean Squares	F ratio
Methods	2	4,317,390	2,158,695	* 19.94
Orthogonal Breakdown of Methods				
Iron vs Con. & Con.-Thin	1		3,860,433	* 35.66
Con. vs Con.-Thin	1		456,960	* 4.22
Within	27	2,923,010	108,259	
Total	29	7,240,400		

Significance Values: F.95 (2,27) = 3.37
F.95 (1,27) = 4.21

Table 6

Analysis of Variance with Orthongonal Comparisons for Solubility Tests

Source of Variation	Degrees of Freedom	Sum of Squares	Mean Squares	F ratio
Replications	7	2.47155	0.35308	* 18.14
Solvents	4	11.75520	2.93880	*150.99
Orthogonal Breakdown of Solvents				
Water vs Acids	1		2.41856	*124.26
Acetic vs Chelators	1		0.66439	* 34.14
Linearity of Chelators	1		8.42165	*432.70
Curvilinearity of Chelators	1		0.25060	* 12.88
Methods	2	0.317136	0.15857	* 8.15
Orthogonal Breakdown of Methods				
Iron vs Con. & Con.-Thin	1		0.27360	* 14.06
Con. vs Con.-Thin	1		0.04354	2.24
Interaction	8	0.11188	0.01398	
Within	98	1.90741	0.01946	
Total	119	16.56340		

Significance Values: F.95 (7,98) = 2.1
F.95 (4,98) = 2.4
F.95 (1,98) = 3.9
F.95 (2,98) = 3.1

From this evaluation it can be seen that the transverse strength of samples produced by the iron-on technic was significantly lower than those produced by either the conventional or conventional-thin technics. Also, the transverse strength of samples produced by the conventional-thin technic was statistically lower than that produced by the conventional technic, but the actual difference was small and probably not clinically significant.

Solubility. The results of the solubility tests were analyzed by analysis of variance, at the 95% level of significance, to test the hypotheses that (1) the three insertion technics possessed the same solubility, and (2) that the five solvents had the same effect on the silicate cement. A significant difference was demonstrated between the three placement technics as well as between the five solvents. This analysis also revealed that the effect of replications was significant, but this factor was removed prior to testing the effects of the solvents and insertion technics. The value for interaction between the rows and columns was not significant. Table 6 contains the statistical model used in this phase of the study.

In order to evaluate the differences illustrated by analysis of variance, the data was further analyzed by orthogonal comparisons. Within the insertion technics the sums of squares value with two degrees of freedom was divided into two parts, with one degree of freedom each, to compare the iron-on technic with the two conventional technics and to compare the conventional with the conventional-thin technic. This evaluation indicated that the silicate specimens produced by the iron-on technic were significantly less soluble than those produced by either the conventional or conventional-thin technics. There was no difference in solubility between the samples produced by the two conventional technics.

Table 7

Ranked Means of the Percent Solubilities
of a Silicate Cement

Method	Solvent	$\bar{X}$ % Solubility
Iron-on	Water	.47
Iron-on	Lactic Acid	.50
Conventional	Water	.56
Conventional	Lactic Acid	.56
Conventional-Thin	Water	.56
Conventional-Thin	Lactic Acid	.60
Iron-on	Acetic Acid	.65
Iron-on	Citric Acid	.76
Conventional-Thin	Acetic Acid	.77
Conventional	Acetic Acid	.80
Conventional-Thin	Citric Acid	.83
Conventional	Citric Acid	.96
Iron-on	EDTA	1.35
Conventional-Thin	EDTA	1.36
Conventional	EDTA	1.46

The sums of squares value, with four degrees of freedom, for the solvents was divided into four parts, with one degree of freedom each, in order to compare (1) distilled water and the four acids, (2) acetic (a nonchelating acid) and the three chelating acids, (3) the three chelating acids for possible linearity from the weakest to the strongest chelator, and (4) the three chelating acids for possible nonlinearity. The results of this comparison indicate that (1) the solubility of the silicate samples produced in this study was significantly less in distilled water than in the acid solvents, (2) the solubility of the samples in acetic acid was significantly less than in the three chelating acids, (3) the solubility of the samples increased very significantly as the ability of the acids to chelate increased, and (4) the increase in solubility with increased chelation ability was not linear but possessed a slight curvilinear trend. Appendix 4 contains the orthogonal mean-square values for the solvents and insertion technics.

The results of the analysis of variance on the solubility tests were further analyzed by Duncan's multiple range test for the purpose of ranking the means. Table 7 contains this data. Values connected with a line are the same at the 95% level of confidence. From this evaluation it can be seen that the percent solubility of the silicate samples was the same in distilled water and lactic acid.

Discussion. The fact that a statistical difference was demonstrated between the transverse strength samples produced by the three insertion technics could be of clinical interest only if the stresses developed in a restoration as a result of oral forces are of such magnitude that only the weaker restorations would be affected. As illustrated in this study, the magnitude of these stresses would have to be between 3,000

and 3,600 psi if a difference were to be seen between the iron-on and conventional-thin technics. In order to see a difference between the conventional and conventional-thin technics, the range of these stresses would have to be between 3,600 and 3,800 psi. Although a statistical difference was illustrated between the transverse strength samples produced by the conventional and conventional-thin technics in this phase of the study, this difference would hardly be considered clinically significant.

The concentration and pH of the acids utilized as solvents in this study were the same. The primary difference between these acids was their ability to chelate. In light of these conditions the effect of chelating agents on silicate cements is quite apparent: as the ability of an acid to chelate increases, the solubility also increases. However, acids which are efficient chelators are not generally present in the oral environment (18).

If it is assumed that the predominant acid in the dental plaque is lactic acid (19) and it is this acid which causes the majority of the solubility of a silicate restoration, then the fact that the solubility of silicate cement is the same in lactic acid and distilled water is of practical interest. The reason for this is that distilled water provides a more stable and convenient solvent to use in the laboratory as well as being the solvent utilized in the present A.D.A. specification test for silicate cement. This result is of further interest in that it depreciates the recent arguments concerning the desirability of using acid media rather than distilled water as a solubility test environment for silicate cements (16, 17).

Figure 6

Photographs of Silver-Plated Impressions
Taken at Seven Days and Three Months

7-Day

3-Months

Iron-On

7-Day

3-Months

Conventional

Clinical Tests

Figure 6 contains representative photographs of the clinical restorations placed with the iron-on and conventional technics. Four individuals evaluated all of the photographs twice. A span of two days occurred between evaluations. Following the examination of each set of photographs of an individual restoration, a decision was made as to whether or not a discernible change had occurred in the surface structure or marginal integrity of the restoration. Following the evaluation the results were tabulated and the percent values of the restorations showing no change in surface and marginal integrity were converted into respective arc sines. These values were then compared by analysis of variance to test the hypothesis that the three insertion technics behaved the same. The arc sine transformations and analysis of variance technics were used in this study instead of the conventional chi square analysis because one of the criteria for the chi square test, namely that not less than 20 percent of the expected values can be less than five, was not met. Table 8 contains the statistical models used in evaluating this data.

The results of this analysis indicated that there was no difference between the three insertion technics and that there was no difference between the two cavity types evaluated (labial and lingual). However, in both the marginal and surface analyses the interaction between cavity types and technics was significant at the 95% level. Also, both analyses indicated that there was a significant difference between observers. However, as the statistical models indicate, the sum of squares values for observers and observer interactions were isolated. This isolation made it possible to test the mean square values of the cavity types and technics with the cavity type times technic interaction mean square.

Table 8

Analysis of Variance for Clinical Tests

Marginal Evaluation

Source of Variation	Degrees of Freedom	Sum of Squares	Mean Squares	F ratio
Cavity Type	1	390.34	390.34	0.83
Technic	2	724.16	362.08	0.77
Cavity Type X Technic	2	938.38	469.19	*51.88
Observers	3	2,571.44	857.14	* 9.48
Observers X Cavity Type	3	140.95	46.98	0.52
Observers X Technic	6	655.51	109.25	1.21
Observers X Cavity Type X Technic	6	542.67	90.44	
Sampling	24	2.127.67		
Total	47	8,091.12		

Surface Evaluation

Cavity Type	1	1.40	1.40	0.002
Technic	2	70.72	35.36	0.05
Cavity Type X Technic	2	1,338.52	669.26	*52.00
Observers	3	1,066.70	355.56	*27.63
Observers X Cavity Type	3	212.87	70.96	* 5.51
Observers X Technics	6	487.29	81.22	* 6.31
Observers X Cavity Type X Technic	6	77.20	12.87	
Sampling	24	1,108.91		
Total	47	4,363.61		

Significance Values: F.95 (1,2) = 18.5
(2,2) = 19.0
(3,6) = 4.76
(6,6) = 4.28

In this manner the effect of differences between observers was removed and consequently did not affect the evaluation of the cavity types or technics.

Discussion. At the time this thesis was prepared a total of 134 restorations had been placed in 25 patients. However, only 62 restorations in 12 patients had been in place for at least three months. Therefore, the results of the clinical phase of this study were based on this limited number of restorations. It is realized that this is a limited number of observations for a clinical study, but the time limitations imposed for completion of this thesis demanded that only this segment of the data be used. This study is to be continued subsequent to the completion of this thesis and the restorations will be recorded at six-month intervals for a period of years.

At three months there were changes present in the surface and marginal integrity of the restorations. However, these changes were limited in number and degree. This limitation offers an explanation for the significant differences seen between observers. It is felt that with an increase in time, the number as well as degree of changes will also increase and that a closer correlation between observers will result.

General Discussion

The results of the laboratory studies indicated that the iron-on technic produced samples which were weaker in transverse strength but less soluble than the conventional method of placing silicate cements. (When the conventional method was used with a thinner mix than normal, there was very little strength loss and no increase in solubility.) If the laboratory tests are true predictors of how silicate cements will behave in the oral environment, then it would be expected that under oral conditions those restorations placed with the iron-on technic would exhibit fewer surface changes due to dissolution in the oral fluids, but at the same time there would be an increased incidence of marginal breakdown due to the reduced edge strength. On the other hand, it is entirely possible that the laboratory tests are far more sensitive than the clinical tests and, because of this, small significant changes in laboratory tests will not be discernible in the clinical environment.

When the seven-day and three-month replications of the clinical restorations were compared, no differences were noted between the three insertion technics used. However, it would be speculative after only three months' service to conclude that these three technics behave the same under oral conditions, for differences may become apparent after six months or a year's time.

It is generally accepted (4, 6, 15, 20) that an inferior silicate restoration will result if less than the optimum amount of powder is used with a given amount of liquid or if the surface is disturbed while the cement is setting. With respect to strength, solubility and clinical performance, a slight increase in the liquid-to-powder ratio did not adversely affect the cement used in this study. Also, the procedure of

disturbing the surface of the setting cement, inherent in the iron-on technic, did not appear to produce an inferior restoration even though the transverse strength was reduced. Once again, these statements are based on a three-month clinical evaluation.

SUMMARY AND CONCLUSIONS

This investigation was designed to determine if the presently used laboratory tests can validly predict the clinical behavior of silicate cements. In order to test for this predictability, three manipulative technics presumed to result in different clinical behaviors were selected. These technics were (1) the conventional matrix-strip technic (a technic long established as being satisfactory), (2) the iron-on (or Olsen) method (presumably producing better adaptation and surface characteristics than the conventional technic), and (3) the conventional technic using a thin mix (presumably producing lower strength and higher solubility than the conventional technic). These three technics were then used to place class III restorations and to prepare samples which were tested for transverse strength and solubility in five different media (water and four organic acids).

As a result of the laboratory phase of this study, it can be implied that the iron-on method of placing silicate cement produces restorations which are weaker in transverse strength but less soluble than those placed with the conventional and conventional-thin technics. The conventional and conventional-thin technics produce restorations with the same solubility characteristics and only slightly different strength values.

Following the three-month clinical evaluation, changes in the marginal and surface integrity of the restorations were observed. However, these changes were not related to the differences demonstrated

by the laboratory tests of strength and solubility. Therefore, on the basis of this three-month clinical study, it can be concluded that the laboratory tests of strength and solubility are not good predictors of clinical performance. On the other hand, with an increase in time, correlative differences may develop.

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APPENDICES

APPENDIX 1

Effect of Mold Temperature on the Transverse Strength of a Silicate Cement Placed with Three Insertion Technics

Prior to starting this investigation the effect of mold temperature on the transverse strength of silicate cement was determined. For this purpose a series of samples was produced with the iron-on, conventional, and conventional-thin technics. The results of this test are presented in the following table.

	Mold Temp.	Insertion Method		
		Iron-On	Conventional	Conv.-Thin
Transverse strength	23° C.	4,200 ± 400	4,300 ± 390	4,100 ± 330
	37° C.	3,000 ± 150	3,800 ± 300	3,600 ± 200

Figures in psi ± $S_{\bar{X}}$ (.95)

n = 10

This data was statistically analyzed as follows:

Source of Variation	Degrees of Freedom	Sum of Squares	Mean Squares	F ratio
Mold Temp.	1	0.846 x 10^7	0.846 x 10^7	* 44.53
Methods	2	0.283 x 10^7	0.142 x 10^7	* 7.47
Orthogonal Breakdown of Methods				
Iron vs Con. & Con.-Thin	1		0.222 x 10^7	* 11.68
Con. vs Con.-Thin	1		0.066 x 10^7	3.47
Interaction	2	0.173 x 10^7	0.086 x 10^7	* 4.53
Within	54	1.021 x 10^7	0.019 x 10^7	
Total	59	2.324 x 10^7		

Significance Values: F.95 (1,54) = 4.0
F.95 (2,54) = 3.1

This evaluation indicated that not only did the iron-on differ from the conventional and conventional-thin methods but that the interaction between the methods and mold temperatures was significant. Because the transverse strengths produced by the three placement methods behaved differently in the two mold temperatures, and the fact that the laboratory tests were being compared with the clinical behavior of silicate restorations placed with the same technics, it was decided to use the mouth temperature mold in this study.

APPENDIX 2

Weight of Residue from 4-ml Samples of Acids Used as Solvents

	Acids			
	Lactic	Acetic	Citric	EDTA
wt. Residue (mg)	.27 ± .08	.18 ± .1	.57 ± .08	1.44 ± .06

n = 10
figures are $\bar{X} \pm S_{\bar{X}}$ (.95)

* * * * * * * * *

Form for Recording Solubility

wt. Bottle No. ____ _____

wt. Wire No. ____ ____

____ ____

Total _____

Tare wt. _____

wt. Bottle + residue _____

wt. Bottle - _____

wt. Residue _____

wt. Solvent Residue - _____

wt. Residue (corrected) _____

Tare + sample _____

Tare wt. - _____

wt. Sample _____

Test:

% Solubility _____

APPENDIX 3

Plating and Photographic Procedures

Silver Plating Elasticon Impressions. The elasticon impressions were stored in individual, two-dram, snap-cap vials until ready for plating. Each vial was numbered and associated with a specific patient and tooth number. Letter designations accompanied each numbered bottle to indicate the time at which the impression was taken (a represented the initial and b the three-month impression).

The following procedure was used to plate the elasticon impressions.

1. Thread a thin copper wire (one strand of household zip cord) through impression so that part of the wire is showing on the surface to be plated. Lightly twist the wire to make fast.

2. Place identification on wire (small piece of colored telephone wire).

3. Cover surface of impression with collodion (a solution composed of two parts collodion, one part amyl acetate and one part ether plus a small crystal of crystal violet). When dry, the collodion film is stripped off and this removes the excess accelerator which accumulates on the surface of the impression during storage.

4. Cover surface of impression with silver powder (Kerr Silver Metalizing Powder). This is applied with a small camel hair brush. The excess powder is removed by air from a chip blower.

5. Paint surfaces which are not to be plated with masking fluid (mimeo correction fluid).

Figure 7

Equipment Used to Photograph
Silver-Plated Impressions

6. Attach three impressions to an alligator clip.

7. Suspend four alligator clips in the plating solution (36 gms AgCN, 60 gms KCN and 45 gms K_2CO_3 in one liter of water).

8. Turn plating machine on, making sure rheostat is in the off position.

9. Advance rheostat slowly until a reading of 150 ma is recorded on the amp meter. (This is equivalent to 12.5 ma per impression.)

10. Plate for one hour.

11. Remove impressions, wash, pour in stone.

Photographing Silver-Plated Replicas. All photographic records for this investigation were made in one series, and the three-month replica of a given restoration was photographed immediately following the original replica. In this manner a close and uniform control was maintained on the procedures of lighting and angulation of the replicas.

The equipment utilized in this procedure is illustrated in Figure 7. A 4 x 5 polaroid film holder was mounted on a Leitz Aristaphot II. A 24 mm., f:4.5 mikro-Summar lens was used with a bellows, extension of 42 cm. This combination produced an enlargement of approximately eighteen times. The replica was mounted on the stage so that a plane tangent to the buccal or lingual margin was parallel to the lens surface. A single light source was directed on the replica at an angle of approximately 15° and perpendicular to the long axis of the crown from the tooth surface toward the restoration. A light meter was utilized to determine the proper exposure. Polaroid Polapan 200, type 52, film was used with a guide number of 30.

APPENDIX 4

Orthogonal Comparisons for Statistical Analysis

Transverse Strength

	ΣX	(1)	(2)
Iron-on	29,368.14	2	0
Conventional	38,489.33	-1	1
Conventional-Thin	35,466.22	-1	-1

	(1)	(2)
Z	-15,219.27	3,023.11
Z^2	231.626×10^6	91.39194×10^5
Div	6 x 10 = 60	2 x 10 = 20
Z^2/Div	3,860,433.0	456,959.7

Total $Z^2/Div = 4,317,392.7$

Source of Variation	Degrees of Freedom	Sum of Squares	Mean Squares	F ratio
Methods				
Orthogonal Breakdown of Methods				
Iron vs Con. & Con.-Thin	1	3,860,433	3,860,433	*35.659
Con. vs Con.-Thin	1	456,960	456,960	* 4.221
Error	27	2,937,010	108,259	

Significance Values: F.95 (1,27) = 4.212

Solubility--Solvents

	ΣX	(1)	(2)	(3)	(4)
H_2O	12.70479	4	0	0	0
Acetic	17.76464	-1	3	0	0
Lactic	13.32390	-1	-1	-1	1
Citric	20.37314	-1	-1	0	-2
EDTA	33.42960	-1	-1	1	1

	(1)	(2)	(3)	(4)
Z	-34.07212	-13.83272	20.10570	6.00722
Z^2	1160.90926	91.34414	404.23917	36.08669
Div	20 x 24 = 480	12 x 24 = 288	2 x 24 = 48	6 x 24 = 144
Z^2/Div	2.41856	0.66439	8.42165	0.25060

Total Z^2/Div = 11.75520

Source of Variation	Degrees of Freedom	Sum of Squares	Mean Squares	F ratio
Solvents				
Orthogonal Breakdown of Solvents				
H_2O vs Acids	1	2.41856	2.41856	*124.26
Acetic vs Chelators	1	0.66439	0.66439	* 34.14
Linearity of Chelators	1	8.42165	8.42165	*432.70
Curvilinearity of Chelators	1	0.25060	0.25060	* 12.88
Error	98	1.907408	0.019463	

Significance Values: F.95 (1,98) = 3.9

Solubility--Methods

	ΣX	(1)	(2)
Iron-on	29.83092	2	0
Conventional	34.81572	-1	1
Conventional-Thin	32.94943	-1	-1

	(1)	(2)
Z	-8.10331	1.86629
Z^2	65.66363	3.48304
Div	6 x 40 = 240	2 x 40 = 80
Z^2/Div	0.27360	0.04354

Total Z^2/Div = 0.31714

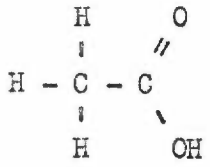
Source of Variation	Degrees of Freedom	Sum of Squares	Mean Squares	F ratio
Methods				
Orthogonal Breakdown of Methods				
Iron vs Con. & Con.-Thin	1	0.27360	0.27360	* 14.06
Con. vs Con.-Thin	1	0.04354	0.04354	2.24
Error	98	1.907408	0.019463	

Significance Values: F.95 (1,98) = 3.9

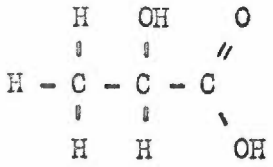
Table 3

Structural Formulas of Acids Used in this Study

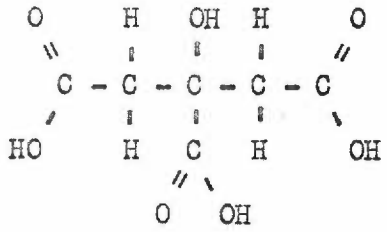
Acetic
CH_3COOH



Lactic
$CH_3CHOHCOOH$



Citric
$(COOH)CH_2C(OH)(COOH)CH_2COOH$



EDTA
$(COOHCH_2)_2N(CH_2)_2N(CH_2COOH)_2$

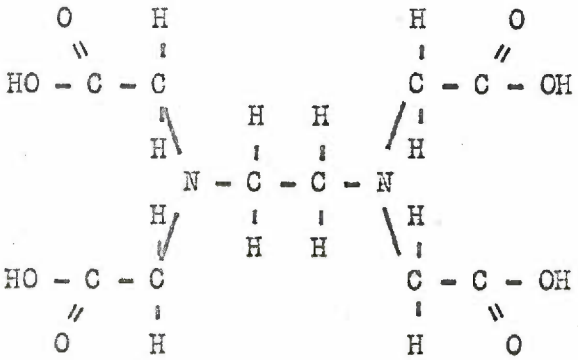


Table 7

Ranked Means of the Percent Solubilities
of a Silicate Cement

Method	Solvent	$\bar{X}$ % Solubility
Iron-on	Water	.47
Iron-on	Lactic Acid	.50
Conventional	Water	.56
Conventional	Lactic Acid	.56
Conventional-Thin	Water	.56
Conventional-Thin	Lactic Acid	.60
Iron-on	Acetic Acid	.65
Iron-on	Citric Acid	.76
Conventional-Thin	Acetic Acid	.77
Conventional	Acetic Acid	.80
Conventional-Thin	Citric Acid	.83
Conventional	Citric Acid	.96
Iron-on	EDTA	1.35
Conventional-Thin	EDTA	1.36
Conventional	EDTA	1.46