NATIONAL BUREAU OF STANDARDS REPORT

9611

Progress Report

on

A LABORATORY AND CLINICAL COMPARISON OF SILICATE CEMENTS AND OF A DIRECT FILLING RESIN, WITH AND WITHOUT FUSED SILICA



U. S. DEPARTMENT OF COMMERCE NATIONAL BUREAU OF STANDARDS

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By

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U. S. DEPARTMENT OF COMMERCE NATIONAL BUREAU OF STANDARDS

A LABORATORY AND CLINICAL COMPARISON OF SILICATE CEMENTS AND OF A DIRECT FILLING RESIN, WITH AND WITHOUT FUSED SILICA

R. L. Bowen, G. C. Paffenbarger and A. L. Mullineaux

Two silicate cements showing a difference of about 0.7 percent solubility and disintegration in distilled water for 24 hours, showed approximately the same durability in clinical service during a four-to-six year period. There was more disintegration in the interproximal areas of the silicate restorations than there was in the self-cleansing areas.

An experimental reinforced resin gave sufficiently good results to warrant further clinical investigations of more recent and perhaps better composite formulations. Both unreinforced and reinforced resin materials appear to benefit from the use of a primer or "cavity seal." A comparison is being made of the clinical durability of two brands of silicate cements. These cements had different solubilities when tested as described in the American Dental Association Specification No. 9 for Dental Silicate Cement.¹ One of these cements, which is referred to as silicate cement "C," had a solubility and disintegration at 24 hours in distilled water of 1.1 percent, over twice that of another cement, which is referred to as silicate cement "D." The primary purpose of the clinical testing was to determine whether this difference in 24-hour solubility in distilled water would be reflected in clinical durability of the silicate cements.

Somewhat later, the project was broadened to include restorations made with a commercially available direct filling resin and a composite material made by mixing the monomer of the direct filling resin with powdered fused silica, which had been treated with a silane coupling agent.

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Both silicate cements (C and D) complied with the requirements of American Dental Association Specification No. 9, which at that time, had the maximum permissible solubility and disintegration at 24 hours in distilled water set at 1.4 percent by weight. This value was reduced to 1.0 percent in second revision of the specification adopted in 1962. Some of the properties of silicate cements C and D are given in Table 1.

The proprietary direct filling resin (Sevriton[®])* consisted of a monomer which was assumed to be predominately methylmethacrylate, an accelerator (<u>para</u>-toluene sulfinic acid or one of its derivatives), and a polymer powder, presumably containing benzoyl peroxide as the initiator for polymerization of the monomer. A "cavity seal" was supplied and recommended by the manufacturer for use in the cavity prior to the insertion of the resin.

*Some commercial materials and equipment are identified to specify the experimental procedure. Such identification does not imply recommendation or endorsement by the National Bureau of Standards, nor does it imply that material or equipment identified is the best available for the purpose.

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The reinforced direct filling resin contained the same proprietary monomer and catalyst. However, in place of the proprietary polymer powder, powdered fused silica was used. This silica powder had been treated with 0.9 percent of vinyltrichlorosilane as a coupling agent in bonding the inert fused silica powder to the resin binder. Benzoyl peroxide (1.2 percent), an initiator for polymerization of the proprietary monomer, was added to the treated silica powder.

Further description of the resin and reinforced resin is given in a previous publication.² Data on a few of the properties of the resin and reinforced resin are given in Table 2.

3. METHODS

Seventy-four silicate cement and twenty-four direct filling resin and reinforced resin restorations were placed, with few exceptions, in proximal surfaces of adjacent teeth as illustrated in Figures 1.A and 2.A.

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One-half of the amount of powder and liquid (0.75 Gm of powder and 0.2 ml of liquid) required to produce a mix of standard consistency of each silicate cement was used in making the mixes. A calcium hydroxide-containing cavity liner was placed on the pulpal walls of most of the cavities prepared for the silicate cement restorations. The cavity seal was used on some but not all of the cavity preparations that were to receive the resin or reinforced resin materials. It was used on both (or neither) of the cavities that were side by side for direct comparison of restorations.

In every case, the matrix was not removed until ten minutes from the time the mix was started. After 24 hours, the fillings were finished down approximately to the margins and polished. The clinical evaluations were made by two dentists soon after the restorations had been dressed down. In some instances, the patients could not return until several weeks after the placements for the finishing and the original inspection.

The evaluations included clinical observations of the surface contour,

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appearance, and color and the conditions of the margins with respect to flushness, apposition, and staining. The rough qualitative degrees of assessment are given in columns 3, 6, 9, 12, 15 and 18, respectively, in Table 3. No common criteria were developed. Instead each observer used his clinical judgment just as if he were an indepent, private practitioner.

Color photographs were taken in duplicate. One or more impressions were made with a poly-sulfide rubber impression material. Permanent casts were prepared using a white "dental stone." The patients were recalled at intervals of about one year, and the restorations were re-evaluated. At the last evaluation (just before this report was prepared), the restorations were four to six years old. At this last examination, each restoration was evaluated in terms of the question, "should this restoration be replaced at this time?." Furthermore, each pair of restorations was evaluated as to which of the two was better in general appearance and condition.

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The summed scores of the two evaluators were, for the most part, converted into percentages. Where this was not feasible, the summed scores were divided by two to correspond to restorations. Fractional numbers are due to different evaluations by the two observers.

4. RESULTS AND DISCUSSION

The qualitative clinical evaluations are given in Tables 3 and 4. The change in the conditions of some of the restorations is shown in Figures 1 and 2.

Surface contour. There was no apparent difference in the loss of substance from the two silicate cements having different solubilities in water. The resins and reinforced resins were definitely better than the silicate cements. There was little, if any, difference between the two types of plastic restorations (columns 3, 4, 5 - Tables 3 and 4).

The amount of solubility and disintegration of each silicate cement restoration varies with the location of different parts of the restoration. As has been pointed out³ the self-cleansing areas have little attack while the areas of the restorations which are not self-cleansing are severely attacked. Some of the loss in the self-cleansing areas may have been caused by abrasion. Fluid exchange with the saliva

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would be at a minimum on those areas of the restorations where severe erosion occurred; these areas were associated with food debris and materia alba. Abrasion and wear would also be minimal in such sheltered areas of the restorations.

Thus the disintegration of the silicate cements resembled dental decay more than a straightforward solubility in the main stream of oral fluids. In other words, the solubility required a very specific and localized biological milieu. Therefore to determine the cause or mechanism of the decomposition of silicate cements in their oral setting, might be as difficult as to determine the mechanism of dental caries.

Surface appearance. After four years, nearly half of the silicates presented roughened surfaces. The resins retained their smooth surfaces, while the surfaces of about 15 percent of the reinforced resins became rough in four years.

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Surface color. Only one shade (21) of silicate cement was used which accounts for the degree of matching as shown by original inspections as given in columns 9 and 10 of Tables 3 and 4. The data show that the cement D with the least solubility in water is not necessarily more resistant to discoloration than cement C with more than twice the water solubility of D (columns 9, 10 and 11 of Tables 3 and 4). Flushness of the margins. There is virtually no difference between the "high" or "low" solubility silicate cement restorations as far as maintenance of the evenness or flushness of the margins. The silicate cements did not seem to hold up as well as the reinforced resin restorations (columns 12, 13 and 14 of Tables 3 and 4). Apposition of the margins. If a dental explorer would catch at the margin when drawn in both directions, this was recorded as a gap or notch at the margin.

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As reflected in columns 15 and 16 in Tables 3 and 4, there was poor apposition in many instances when the restorations were evaluated the first time. The situation went from bad to worse in time with the silicate cements. The reinforced resin restorations had the best ranking with respect to apposition over the time period observed.

Staining at the margins. All of the silicate cements and the resins showed a tendency to become somewhat stained at the margins during four years of service in the mouth (columns 18, 19, 20 in Tables 3 and 4).

When the proprietary cavity seal was used as directed by the manufacturer, there appeared to be relatively better adaptation and less staining of the margins. The clinical impression was that this was the case with both the resin and reinforced resin materials. Length of serviceability. The question "Should this restoration be replaced at this time?" was asked at the final inspection. The tabulated replies to this question are given in Table 5. Sixty-four restorations of silicate cement and twenty-four resin restorations were available for the final inspection. About 42 per cent of the silicate cement restorations needed replacement in from 3-1/2 to 6-1/2

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years. Cement C ("high solubility") had as good a length of service as coment D ("low solubility"). The resin and reinforced resin restorations had about 4 per cent that needed replacement after 4 to 5 years. No recurrent decay was detected at the margins of any of the restorations when the teeth were observed with transillumination. However, restorations were not removed to see if there was underlying decay.

<u>Comparison of restorations</u>. A comparison of the general appearance of paired restorations was made at the last examination, where the restorations were in close proximity and of similar size and type. More of the silicate cement C ("high solubility") restorations were judged in better condition compared with cement D ("low solubility") restorations (Table 6). This seeming inconsistency between the "high" and "low" solubility silicate cement restorations is also reflected slightly in some of the data given in Tables 3 and 4.

In the small number of the resin and reinforced resin restorations that were compared after 4 to 5 years, there was little difference in appearance (Table 6).

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5. SUMMARY AND CONCLUSIONS

Two silicate cements showing 1.1 and 0.4 percent solubility and disintegration in distilled water for 24 hours, when tested as described in American Dental Association Specification No. 9 for Dental Silicate Cement, did not show corresponding durability in clinical service. Restorations of neither of the silicate cements showed much loss of material in self-cleansing areas during 4 to 6 years of service, rather, they disintegrated in an interproximal, decay-like manner.

A reinforced resin gave sufficiently good results to warrant further clinical investigations of more recent and perhaps better composite formulations. Both unreinforced and reinforced resin materials appeared to benefit from the use of a primer of "cavity seal."

No recurrent marginal decay was detected on any of the restorations with the aid of transillumination. No restorations were removed to inspect for underlying decay.

About 42 percent of the silicate cement restorations were judged to need replacing after 5 \pm 1-1/2 years. On the other hand only about 4 percent of the resin and reinforced resin restorations needed replacement after 4 \pm 1 years.

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REFERENCES

- American Dental Association Specification No. 9 for Dental Silicate Cements. <u>Guide to Dental Materials</u> (1962-1963), American Dental Association, Chicago, Illinois, page 54.
- Bowen, R. L. Effect of particle shape and size distribution in a reinforced polymer. JADA 69:481 Oct., 1964.
- 3. Henschel, C. J. Observations concerning in vivo disintegration of silicate cement restorations. J Dent Res 28:528 Oct., 1949.



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Cement	Powder	Setting time	Compressive strength	Solubility and disintegration	Opacity	Arsenic content
	Gm*	minutes	kg/cm ²	%	Co.70	%
c†	1.55	9	1,710	1.1	45-50	<0.0002
Dţ	1.50	4	2,100	0.4	45-50	<0.0002
ADA Sheri-		min max			min max	max
fication No. 9 for	*	® M	1,620	1.4	35 55	0.0002
Dental Silicate Cement	*	00 	1,700	1.0	35 55	0.0002
* Grams of powd standard cons	ler necessa istency (d	ry to mix v isk 25 ± 1	vith 0.4 ml of mm in diameter	liquid to produ	ce a mix of	-
t Tests were co Cement First	nducted as Revision (described 1950-1961)	in ADA Specifi	cation No. 9 fo	r Dental Si	licate

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First revision of specification (1950-1961)

+ Second revision of specification (1962). Same as FDI Specification No. 5 for Dental Silicate Cement

TABLE 1

TABLE 2

Properties of a direct filling resin with and without mineral reinforcement

Material	Resin content*	Time of hardening†	Compressive strength	Modulus of elasticity	Opacity‡	Coefficient of thermal expansion
	%	minutes	kg/cm ²	kg/cm ²	Co . 7 0	ppm/°C
Direct filling resin A+	7.66	9	850	21,000	45	100
Direct filling resin A rein- forced with fused silica particles+	26-30	4	1,950	98, 500	20	26
* Percentage by	/ weight o	f material vo	olatile at 700	°C		
t Interval betv	veen the b	eqinning of t	the mixing and	the time wh	nen a stand	lard

- for Gillmore needle failed to make a perceptible circle on the surface of the specimen. Test was conducted as described in FDI Specification No. 5 Dental Silicate Cement
- # Test conducted as described in ADA Specification No. 9 for Dental Silicate Cement
- + Complied with color stability test in FDI Specification No. 3 for Denture Base Polymer



TABLE 2

Properties of a direct filling resin with and without mineral reinforcement

Material	Resin content*	Time of hardening†	Compressive strength	Modulus of elasticity	Opacity‡	Coefficient of thermal expansion
	%	minutes	kg/cm ²	kg/cm ²	Co • 7 0	ppm/°C
Direct filling resin A+	69.7	Q	850	21,000	45	100
Direct filling resin A rein- forced with fused silica particles+	26-30	4	1,950	98, 500	20	26
* Percentage b	y weight o	f material vo	olatile at 700	C °		
<pre>t Interval bet Gillmore need</pre>	ween the b dle failed	eginning of t to make a pe	the mixing and	the time where solutions that the second sec	nen a stand surface of	lard the

Test conducted as described in ADA Specification No. 9 for Dental Silicate Cement

specimen. Test was conducted as described in FDI Specification No. 5 for

Dental Silicate Cement

+ Complied with color stability test in FDI Specification No. 3 for Denture Base Polymer

								Table	3										
		·		Clinic	cal characte	eristics of	silicate	cement and	plastic ro	storation	ns after four	years of	scrvice						
1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20
Material	Number of restorations at 4 ycars	Surf	ace contou	ır	Sur	face appcar	ance		Color		Flushne	ss of marg	ins	Appositi	on of marg	ins	Margi	nal stainir	ng
		condition	original	4 years later	condition	original	4 years later	condition	original	4 years later	condition	original	4 years later	condition	original	4 years later	condition	original	4 years later
		over	% 10	% 3		%	%	lighter	% 30	% 11	over	% 4	% 0		%	%	none	% 90	% 49
Silicate cement C solubility	35	normal slightly	77	34	smooth	94	54	good	61	66	éven	86	24	touching	90	41	trace	7	27
1.1%		under severcly	12	20	rough	6	46	darker	9	23	under	10	76	_gap	10	59	hcavy	0	3
		over	13	7					1										
Silicate cement D	20	normal	77	37	smooth	97	54	lighter	17	1	over	4	4	touching	81	45	trace	6	28
0.4%		under	10	32			[good	73	59	even	87	29	gap	19	55	slight	1	19
		severely under	0	24	rough	3	46	darker	1.0	40	under	9	67				heavy	3	16
Direct filling	11	over normal	9 73	0 73	smooth	95	100	lighter	9	0	over	23	0	touching	91	77	none	86	36
resin A		slightly	18	27					02	69		77	77		9	23	trace	5	32
		severely	0		rough	5	0		02	00	even			gup			heavy	0	9
		under						darker	9	32	under	0	23		.				
Direct filling		normal	77	65	smooth	100	85	lighter	38	27	over	16	8	touching	81	85	none	15	42
resin A rein-	13	slightly	15	27									<u> </u>		19	15	slight	8	27
forced with fused silica particles		under severely					10	good	62	61	cven	69	69	Jap	1	10	heavy	0	0
-		under	0	0	rough	0	15	darker	0	12	under	15	23				1	<u>n</u>	<u>!</u>





Table 4

1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20
Material	Number of restorations at 5 years	Su	rface cont	our	Surf	ace appear	ance		Color		Flushne	ss of marg	ins	Apposit	ion of mar	gins	Margin	al staining	a
	40 5 700-5	condition	original	5 years later	condition	original	5 ycars later	condition	original	5 years later	condition	original	5 years later	condition	original	5 years later	condition	original	5 years
		over	%	%		%	%	lighter	% 36	% 23	over	% 0	% 6		%	%	none	%	%
Silicate cement C	26	normal	83	35	smooth	96	54	good	60	44	even	90	21	touching	83	52	trace	6	33
solubility 1.1%	20	under	9	34				darker	4	33	under	10	73	gap	17	48	slight heavy	4	21
		under	2	27	rough	4	46												
Silicate cement D		normal	77	45	smooth	95	66	lighter	23	4	over	5	5	touching	84	46	none	84	36
solubility 0.4%	28	slightly under	12	25				doog	77	46	even	86	27	gap	16	54	trace	9	23
		severely under	2	28	rough	5	34	darker	0	50	under	9	68				heavy	7	16
Direct filling	2	over normal	0 25	0 50	smooth	100	100	Lighter	50	50	over	0	0	touching	75	100	none	100	25
resin A		slightly	50	25				good	25	25	even	100	75	gap	25	0	trace	0	50 25
		severely	25	25	rough	0	0	darker	25	25	under	0	25				heavy	0	0
		over	0	0	smooth	100	100	lighter	60	0	over	0	20	touching	80	90	none	50	60
Direct filling resin A rein-	5	slightly	20	40	Smooth				40	80	even	80	40	qsp	20	1.0	slight	30	30
forced with fused silica particles		under severely	0	0	rough	0	0	dankan		20	undor	20	40	- Ack	1 20		heavy	0	0
		under			1	1		uarker	0	20	under	20	40					4	

Clinical characteristics of silicatecement and plastic restorations after five years of service



TABLE 5

Length of serviceability

Age of Restoration	Material	Replacen Number of Res	ment storations*
Years	Silicate Cement	No	Yes
$4 \pm \frac{1}{2}$	<u>С</u> 	4 5	2 3
5 ± 🛓	C D	12 12.5	10 10.5
6 ± 불	C D	1 2.5	1
тс	TAL RESTORATIONS	37	27
	Resin	7.5	0.5
4 ± 호	Reinforced resin	10	0
5 ± 支	Resin Reinforced	3	0
TC	TAL RESTORATIONS	23	1

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* Summed scores of the two evaluators divided by two

TABLE 6

Comparisons of paired restorations

Silicate cement	C is better than D	Number of restorations* 16.5
restorations	C and D are about the same	6.5
old	D is better than C	8.5
	Unreinforced resin is better than reinforced resin	3.5
Resin restorations	Unreinforced resin and rein- forced resin are about the	3.5
4-5 years old	same Reinforced resin is better than unreinforced resin	3

* Summed scores of the two evaluators divided by two, resulting in fractional numbers



Fig. 1.A.: Class III restorations soon after they were placed. In the mestal of the upper left cuspid, silicate cement was used with a calcium hydroxide-containing liner; in the distal of the lateral incisor, a direct filling resin was placec following the application of a cavity seal. In the mestal of the lateral incisor is a reinforced resin restoration and in the distal of the central incisor is a direct filling resin, in both of which a cavity seal was used.







Fig. 2.A.: Class III restorations in the mesial surfaces of central incisors soon after they were placed. In the patient's left incisor is silicat cement D. In the patient's right incisor is a reinforced resin. A calcium hydroxide-containing liner was used under each restoration.





