AN EVALUATION OF FACTORS INFLUENCING MARGINAL LEAKAGE OF AMALGAM USING AN AIR PRESSURE METHOD - III

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ABSTRACT

The relationship of factors influencing the in vitro marginal leakage of dental amalgams was investigated. The factors included were alloy type, precondensation and postcondensation mercury content, as well as the difference between them, lateral setting dimensional change, and packing factor. Using a stepwise-linear multiple regression analysis, a significant relationship could not be established between these independent variables and either leakage or dimensional change as the dependent variable.

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Introduction and Literature Review

This paper is a continuation of the research conducted within the Dental Materials Department at the University of Oregon Health Sciences Center-School of Dentistry concerning those factors that influence the in vitro marginal leakage of amalgam using an air pressure method. This paper is specifically predicated on the results and conclusions reached in the most recently completed such study by Tantiniran. (13)

Her conclusions were as follows:

1. Within each alloy tested, as the plasticity of the precondensation mix was increased--
   a. leakage decreased
   b. lateral setting dimensional change increased markedly
   c. axial setting dimensional change increased only slightly
2. Across alloys, leakage was significantly different--
   a. at the same plasticity
   b. at the same lateral dimensional change
3. The nature of the alloy particles appeared to influence the observed leakage. The spherical particle alloys investigated in this study showed more leakage than alloys having chip-cut or dispersant blend particles.

These conclusions for the most part are in agreement with the other research literature in this area. (2, 3, 5, 7, 8, 10, 11, 13, & 14) The notable exception to this agreement involves the relationship between plasticity and leakage. (7) This problem will be addressed below.

Evaluations of the marginal adaption of dental amalgams have been conducted using several criteria. (9) These include radioisotope or dye penetration of the margin interface, visual or electronmicroscopic observation of the marginal seal, and measurement of the leakage of high pressure gas through the marginal area. (9) The use of dyes and radioisotopes results in subjective results and does not allow the retesting of any given sample. Visual determinations are time consuming, require elaborate equipment, and allow only the evaluation of a sectional view of the interface area.

The air pressure test system described by Granath in 1970 (6) has the advantages of delivering quantified results and allows the retesting of any sample. This retesting capability becomes invaluable when investigating the influence of extrinsic factors such as corrosive agents, thermal cycling, or even time on leakage.

Hackman, in 1976, constructed a modified, Granath type air pressure test apparatus. (8) He established the reliability of this system which was used by Biermann and Tantiniran in subsequent studies. (3, 13)

Comparing results and conclusions of the many papers dealing with marginal leakage is fraught with problems. Two of the most important problems are the lack of consistent test parameters and definition of terms.

The term plasticity has caused a great deal of confusion in the literature. The plasticity or wetness of a mix of any given alloy is a direct function of the precondensation mercury/alloy ratio; as the mercury content increases, so does the plasticity of the mix. The problem arises when comparing mixes of different alloys. Tantiniran used a smear test to arrive at a constant, "ideal" plasticity of her test samples. This test is obviously dependant on the subjectivity of the operator. Granath defines plasticity as the difference between precondensation mercury content and postcondensation mercury content of an amalgam sample. (7) The validity of his conclusion that leakage is inversely related to plasticity is doubtful in view of the fact that samples with the same plasticity had leakage values that ranged from the highest to the lowest observed in his study.

There is good agreement that most alloys undergo a negative dimensional change during the setting reaction, ie: they contract. (3, 7, 8, 10, 11, 13, & 14) These same authors were not able to establish a significant relationship between axial dimensional change and leakage within or across alloys. Vrijhoef and Tantiniran were

able to confirm that as less lateral contraction occured during setting within alloys, better marginal adaption (14) and less leakage (13) was observed. They could not establish this relationship across alloys pointing to alloy type as an important, independant factor. Noting that both Vrijhoef and Tantiniran observed lateral dimensional changes different than axial dimensional changes for any given sample brings up the possibility that setting dimensional changes may be a surface phenomenon.

Koran and Asgar investigated the relative leakage of three spherical alloys using a high pressure nitrogen system. (10) The three alloys differed in the relative size of their particles. They observed a significant difference in leakage between the alloy comprised of particles less than 44 microns and the other two alloys. Mahler, when studying the microstructure of set amalgams, was able to detect a difference in original alloy particle size. (12)

Because of the consistent inability to establish relationships of various factors across alloys, one must consider some factor or factors inherent to the alloy itself. In the search to identify such a factor, it was noted that equal masses of different alloys appeared to occupy different volumes, ie: they had different apparent densities. Being of similar chemical composition, all alloys should have similar true densities. It was decided to investigate the difference between the true and

apparent densities of selected test alloys. The difference between these two densities could be a reflection of the particle shape and/or particle size distribution in the alloy powder. These physical parameters of the alloy particles would affect how well or close the particles pack together. The closer the particles fit together, the higher the apparent density should be.

Densities are stated in units of mass per volume. The recipricol of a density would be in units of volume per mass. Subtracting the recipricol of the true density from the recipricol of the apparent density will result in the measure of interparticle space associated with a given mass of alloy. This difference will be called the "packing factor" of the alloy.

The objectives of this investigation were to:

1. Establish a protocol for determining the true and apparent density of an alloy and using these, determine the packing factor of that alloy.
2. Determine the following factors for each of eight alloys:
   a. packing factor
   b. plasticity of mix
   c. relative marginal leakage
   d. lateral setting dimensional change
3. Establish the relative influence of these factors on marginal leakage using a stepwise-linear multiple regression analysis.

Materials and Methods

Alloys selected for evaluation. Based on previous studies, eight alloys were selected that would provide a relatively large range of the influencing factors to be considered. The spherical alloys have consistently exhibited high leakage values when tested using an air pressure system. The conventional alloys have generally fallen in the midrange, while the dispersant blends have allowed minimal leakage. The alloys selected for evaluation are listed below:

Spherical

Tytin, S. S. White, batch #967902

Shofu, zinc free, Shofu, batch #1057

Spheraloy, Kerr, batch #T155

Caulk Spherical, L. D. Caulk, batch #W3-26

Conventional

New True Dentalloy, S. S. White, batch #01387312

Optaloy, L. D. Caulk, batch #30E70

Speyer, Speyer Smelting and Refining, batch #7702

Dispersant Blends

Dispersalloy, American Silver and Mercury, no batch #

Marginal leakage test system. The leakage test apparatus and protocol used for this project was the same as that used by Hackman, Biermann, and Tantiniran. (3, 8, & 13) This system was constructed and test protocol developed by Hackman. It is a modified version of a system originally designed and described by Granath and Swenson. (6) The major modifications made by Hackman were the deletion of the flowmeter and the temperature control system. By monitoring both the high pressure air line temperature and room temperature and testing only when both were equal to a constant 23° C., temperature was eliminated as an influencing factor. The results of Hackman's reliability tests with this system indicated the flowmeter was not a necessary component.

This test system allows a constant, predetermined air pressure, 300 mm Hg, to be applied to one side of an amalgam sample and sample holder and the collection of any air that is able to penetrate the margin between the amalgam and the sample holder. Any air that penetrates constitutes marginal leakage. This air is collected and measured using water displacement in a graduated burette. By observing the amount of air penetration over a constant time span while keeping air pressure, air and room temperature, and the amalgam/sample interface area constant, one can derive a numerical index of relative marginal leakage. The burette in which the

leakage air was collected and measured was graduated in units equal to 0.05 ml. The leakage air was collected for two minutes for each test run, as was done in previous studies. (3, 8, & 13) The index of relative marginal leakage is thus stated in 0.05 ml units/two minutes. Without exception, it was noted that on each test the volume of leakage in 0.05 ml units was equal to the number of bubbles observed in the two minute test period, ie: each bubble equals 0.05 ml. This relationship is coincidental to the test parameters used in this study.

Leakage test sample holders. The amalgam sample holders consisted of one inch diameter by 4.5 ± 0.1 mm thick discs of 902 precision machinable ceramic from Cotronics Corporation. In the center of these discs, 4.50 mm holes were drilled to simulate class one cavities. Each holder was cleaned with methanol and air dried to remove any machining debris, then marked to designate the alloy and specimen number on top of the disc. The discs were fitted into a recessed, plastic mold to provide a bottom to the cavity when condensing the amalgam.

The following mixing parameters were used in the preparation of the amalgam samples tested for leakage.

Trituration time. Each alloy was tested for proper trituration time by using a slightly dry mix. (13) Immediately following an initial trituration time of

3 seconds, the mix was rolled into a ball in the palm of the hand. Trituration time was increased in 2 second intervals until the mix resulted in a smooth, homogeneous, bright ball without fissures. This time was taken as the proper trituration time.

Precondensation mercury content. Trial mixes were prepared for each alloy using the same alloy/mercury ratios as were used for the trituration tests. These were condensed into the ceramic discs. If free mercury was not expressed during the condensation procedure, the mercury percentage was increased in one percent increments until excess mercury was observed. This alloy/mercury ratio was used to prepare all the samples tested in this study. The selected mixing parameters for each alloy are presented in table 1.

Condensation procedure. To standardize the condensation technique, all specimens were condensed by one operator. Hand condensation was accomplished with a 2 mm diameter smooth face condenser. A condensation force of $2\frac{1}{2}$-$3\frac{1}{2}$ pounds was monitored by mounting the recessed, plastic condensation platform on a calibrated sonic force measuring transducer. This device provides a low and high pitch sound at the low and high load range respectively. Each specimen was prepared using five increments of amalgam with the same amalgam carrier and 12-16 vertical strokes per increment. All samples were condensed within $2\frac{1}{2}$-3 minutes from the end of

trituration. Excess amalgam was carefully carved from the top and then the bottom of the sample using a sharp razor blade. The blade was moved from the edge toward the center of the restoration to prevent packing the margins with carving debris.

Storage conditions. Specimens for the leakage tests were stored in air in covered crystallizing dishes for 48 hours to allow completion of the setting reaction.

Air pressure leakage test. Using the protocol established by Hackman (8), five samples of each of the eight alloys were tested for leakage. The results are presented in table 2.

Dimensional change. Lateral setting dimensional changes were measured on all eight amalgams. Five samples of each amalgam were prepared mechanically according to A.D.A. specification number 1 for amalgam. The mixing parameters were as listed in table 1. This specification results in a machine packed cylinder of amalgam 4 mm in diameter and approximately 8 mm long. The change in diameter of the test cylinders from 5 minutes after completion of trituration to 24 hours was measured with a recording transducer. The results of these tests are presented in table 3. The results are presented in microns of change rather than microns per centimeter because at this time we cannot be sure that the dimensional change is uniform across the diameter. If dimensional change were a surface phenomenon, it would be independent of specimen size.

Residual mercury/plasticity. Residual mercury content was determined for each of the 40 leakage samples using the procedure described by Crawford and Larsen. (4) This involves heating the amalgam sample in an inert atmosphere at 1000° F. for one hour. Our inert atmosphere was argon rather than the nitrogen used by Crawford. This results in the mercury contained in the specimen being boiled off, with a subsequent loss in weight. Utilizing this weight loss, a residual mercury content can be determined. By comparing the residual mercury with the precondensation mercury, the difference can be calculated. This change would be called plasticity by Granath. (7) The initial mercury content, the final mercury content, and the difference is presented in table 4.

Apparent density. The apparent volume for this determination was obtained by pouring different amounts of alloy powder into a glass tube sealed on one end with a glass cover slip. The glass tube was vibrated with a small hand vibrator for successive periods of 30 seconds to settle the powder. At the end of each 30 second period, the height of the alloy column was measured with a steel millimeter rule to the nearest 0.25 mm. When the column reached a steady state, ie: the height did not change from the last reading, this height was used to calculate the volume of alloy in the tube.

The total vibration time required to reach a steady state ranged from 3 minutes to 5 minutes.

A section of the glass tubing used to make the measuring device was cross-sectioned at three levels and the inside diameter was determined using a measuring microscope. The inside diameter was 3.967 ± .017 mm. By knowing the height and diameter of a cylindrical column of alloy, the volume can be determined. The mass of each sample was determined by weighing the glass tube before and after filling with alloy. The apparent densities of 5 samples of each of the eight alloys was determined in mg/mm^3. This is equivilent to gm/cc and is presented as such in table 5.

True density. The true volume values used to determine the true density of an alloy sample were obtained using a Beckman model 930 air comparison pycometer, Beckman Instruments, Inc. The standard operation mode, 1 to 2 atmosphere operation as described in paragraph 5.2 in the instruction manual was used for all measurements. This device measures the volume of powdery, granular, porous, or irregularly-shaped solids utilizing the principle of gas displacement. Room air was the gas used in this study. Five samples of each alloy were measured. The mass of each sample was determined by weighing the sample container before and after filling it with alloy. The true densities of the eight alloys are presented in gm/cc in table 5.

Packing factor. The packing factor for each alloy was calculated using the following formula:

AD = Apparent density in gm/cc

TD = True density in gm/cc

P.F. = Packing factor in cc/gm

$$1/AD - 1/TD = P.F.$$

Table 1.

AMALGAM SAMPLE PREPARATION CRITERIA

Alloy	Hg %	Pestle	Mixing Time
NTD #01387312	50	steel	9 secs
Spheraloy #t155	48	steel	9 secs
Dispersalloy # unknown	50	steel	10 secs
Tytin #967902	42	steel	9 secs
Shofu #1057	45	steel	9 secs
Speyer #7702	51	steel	9 secs
Optaloy #30E70	53	plastic	9 secs
C. Spherical #W3-26	46	plastic	11 secs

Notes:

1. All samples were prepared using 600 mg of alloy in powder form.

2. Trituration was accomplished using a Caulk Varimix amalgamator set at the M2 position.

3. All samples were mixed in a J & J plastic, friction-lock capsule.

4. A cylindrical steel pestle was used for all mixes except the two Caulk alloys, Optaloy and Caulk Spherical. As per Caulk recommendations, a plastic dumbbell shaped pestle was used for those alloys.

Table 2.

LEAKAGE VALUES (.05 ml/unit)

Alloy	n	Hg %	$\bar{X}$	S.E.
C. Spherical				
Observed	5	46	3.2	.49
Log 10			.49	.063
NTD				
Observed	5	50	3.8	.20
Log 10			.57	.026
Dispersalloy				
Observed	5	50	6.2	.58
Log 10			.78	.039
Speyer				
Observed	5	51	8.0	1.92
Log 10			.86	.098
Optaloy				
Observed	5	53	13.5	2.32
Log 10			1.11	.080
Tytin				
Observed	5	42	14.4	.40
Log 10			1.16	.014
Shofu				
Observed	5	45	46.0	8.50
Log 10			1.63	.089
Spheraloy				
Observed	5	48	60.0	5.01
Log 10			1.77	.037

Notes:

1. Leakage values were observed using a constant 300 mm Hg air pressure at 23° C. and over a two minute observation period.

2. Alloys connected with vertical lines are not significantly different using Scheffe's contrast following ANOVA (p=0.05).

Fig. 1.

Log 10 leakage values vs alloy type

Alloys connected with horizontal lines are not significantly different using Scheffe's contrast (p=0.05) following ANOVA.

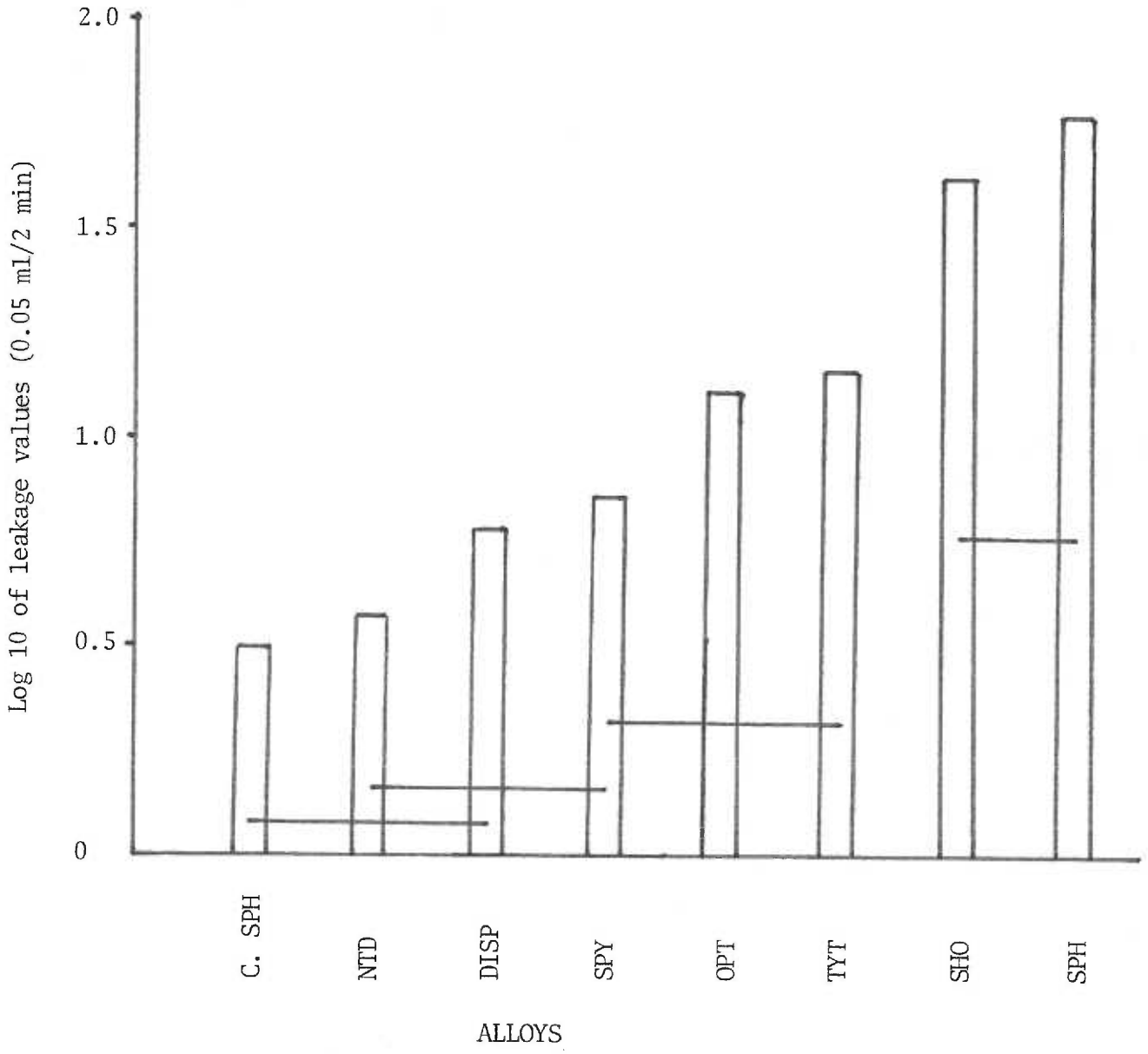

Table 3.

LATERAL DIMENSIONAL CHANGE

Alloy	n	Hg %	$\bar{X}$	S.E.
C. Spherical	5	46	- 3.0	.256
Tytin	5	42	- 4.2	.278
Dispersalloy	5	50	- 5.1	.459
Shofu	5	45	- 7.5	.516
NTD	5	50	- 9.8	.847
Optaloy	5	53	-11.9	.568
Spheraloy	5	48	-12.7	.500
Speyer	5	51	-13.1	.554

Notes:

1. $\bar{X}$ is in microns of observed change.
2. Change is from 5 minutes after completion of trituration to 24 hours.
3. Alloys connected with vertical lines are not significantly different using Scheffe's contrast following ANOVA (p=.05).

Fig. 2.

Lateral dimensional change in microns vs alloys

Alloys connected by horizontal lines are not significantly different using Scheffe's contrast (p=0.05) following ANOVA

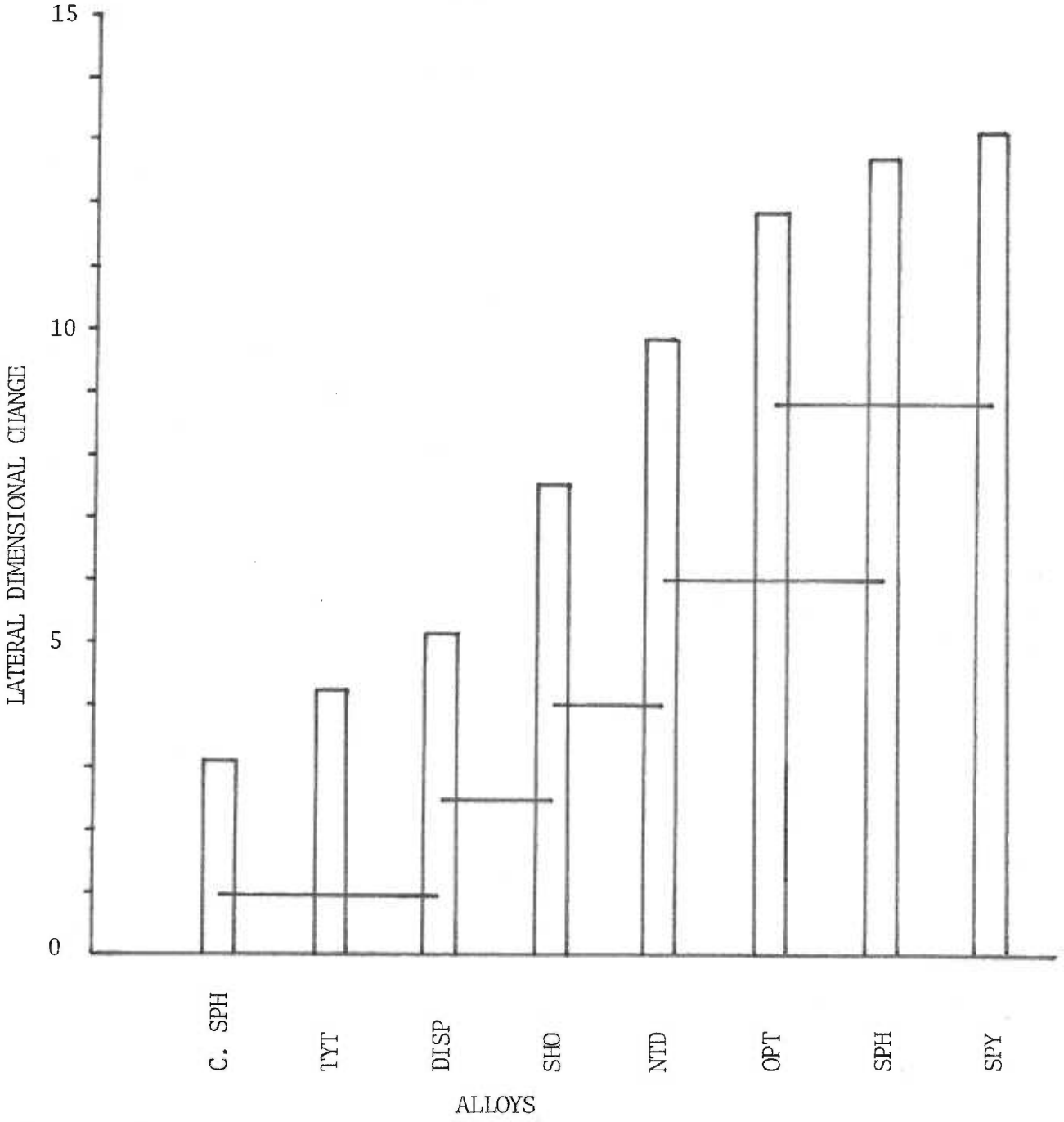

Table 4.

HG CONTENT ANALYSIS

Alloy	n	Initial	Final $\bar{X}$ (S.E.)	Difference
OPT	5	53 %	52.0 (.049)	1.0
SHO	5	45	43.6 (.113)	1.4
SPH	5	48	46.4 (.142)	1.6
C. SPH	5	46	44.4 (.111)	1.6
TYT	5	42	40.2 (.112)	1.8
DISP	5	50	46.9 (.353)	3.1
SPY	5	51	47.7 (.169)	3.3
NTD	5	50	46.4 (.083)	3.6

Table 5.

ALLOY DENSITIES gm/cc - TRUE (TD) AND APPARENT (AD)

Alloy	n	TD $\bar{X}$ (S.E.)	AD $\bar{X}$ (S.E.)
TYT	5	9.84 (.016)	6.68 (.022)
SHO	5	10.04 (.024)	6.36 (.011)
SPY	5	10.08 (.034)	3.19 (.031)
SPH	5	10.09 (.029)	6.35 (.019)
NTD	5	10.11 (.018)	5.13 (.008)
DISP	5	10.13 (.026)	5.70 (.013)
C. SPH	5	10.13 (.036)	5.50 (.016)
OPT	5	10.16 (.026)	4.32 (.022)

Note: True densities of alloys connected with the vertical line are not significantly different using Scheffe's contrast following ANOVA (p=.05).

Table 6.

PACKING FACTOR cc/gm

Alloy	n	$\bar{X}$	S.E.
TYT	5	.048	.0005
SHO	5	.058	.0004
SPH	5	.058	.0004
DISP	5	.077	.0004
C. SPH	5	.083	.0005
NTD	5	.096	.0005
OPT	5	.133	.0012
SPY	5	.215	.0030

Note: Alloys connected with vertical lines are not significantly different using Scheffe's contrast following ANOVA (p=.05).

Table 7.

MEANS OF FACTORS

Factor	L	L10	D	P.F.	IM	M	FM
Units	.05ml/2min	.05ml/2min	Microns	cc/gm	%	%	%
C. SHP	3.2	.49	- 3.0	.083	46	1.6	44.4
NTD	3.8	.57	- 9.8	.096	50	3.6	46.4
DISP	6.2	.78	- 5.1	.077	50	3.0	47.0
SPY	8.0	.86	-13.1	.215	51	3.3	47.7
OPT	13.5	1.11	-11.9	.133	53	1.0	52.0
TYT	14.5	1.16	- 4.2	.048	42	1.8	40.2
SHO	56.0	1.63	- 7.5	.058	45	1.4	43.6
SPH	60.0	1.77	-12.7	.058	48	1.6	46.4

Note. L= observed leakage; L10= Log base 10 of observed leakage; D= lateral setting dimensional change; P.F.= packing factor; IM= initial mercury content; M= loss of mercury during condensation; FM= final (residual) mercury content.

Table 8.

CORRELATION COEFFICIENT MATRIX

D	-.35				
PF	-.35	-.58			
FM	-.11	-.65	.59		
M	-.55	-.11	.39	.04	
IM	-.26	-.66	.67	.96	.31
	L10	D	PF	FM	M

Note: L10=base 10 Log of leakage; D=dimensional change; PF=packing factor; IM=initial Hg; M=change in Hg; FM=final Hg

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Table 9.

ANALYSIS OF VARIANCE
L with M, D, IM, & PF

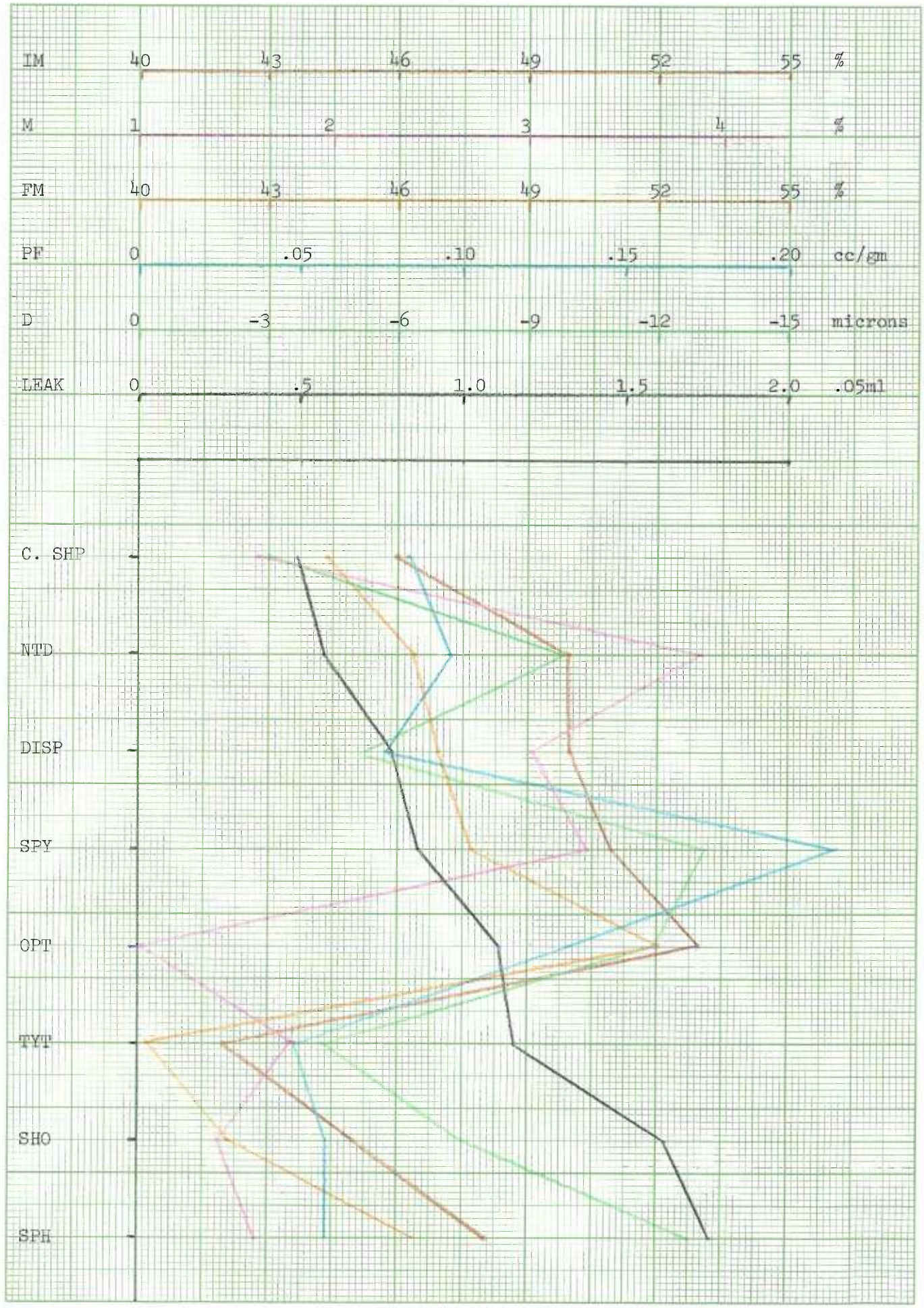
Source	DF	SS	MS	F
Regression	4	1.230	.308	3.149 n.s.
Residual	3	.293	.098	

Table 10.

ANALYSIS OF VARIANCE
D with PF, M, & IM

Source	DF	SS	MS	F
Regression	3	55.323	18.441	1.280 n.s.
Residual	4	57.637	14.409	

n.s. = not significant


IM
40
43
46
49
52
55
%
M
1
2
3
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FM
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49
52
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D
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NTD
DISP
SPY
OPT
TYT
SHO
SPH
FACTORS VS ALLOYS

Figure 3.

RESULTS AND DISCUSSION

The air pressure test system built by Hackman (8) proved again to be highly reliable. Using eight random double determinations, a standard error of the measure of 1.1 unit was calculated. The observed leakage values are depicted in table 2 in ascending order. Due to non-homogeneity of variances, (small variances associated with small means and large variances associated with large means), a base 10 log transform was performed on the leakage data prior to statistical analysis. Analysis of variance followed by the use of Scheffe's contrast was used to compare differences among alloys. Figure 2 is a bar graph representation of the calculated differences.

Tytin, Shofu, and Spheraloy, all spherical alloys, exhibited the most leakage as was expected from previous investigations. (3, 8, & 13) It is interesting to note that the group exhibiting the least leakage, Caulk Spherical, New True Dentalloy, and Dispersalloy, includes a spherical, a conventional, and a dispersant blend alloy respectively. This information negates the possibility of predicting relative marginal leakage based on alloy particle type alone.

All the alloys evaluated for lateral setting dimensional change contracted in the period from 5 minutes to 24 hours following trituration, see table 3.

There was a range of absolute dimensional change of 10 microns, ie: -3.0 μ to -13.1 μ, but no consistent relation to alloy type could be established, see figure 2. As all the dimensional change specimens were mechanically prepared cylinders, 4 mm in diameter, the dimensional change data could have been presented as a change in diameter compared to the diameter of the specimen, ie: microns per centimeter, without affecting the results of this investigation. It was not presented as such and should not be until setting dimensional change, both lateral and axial, can be proven not to be surface phenomenon.

Determination of the true densities of the alloy powders confirmed the expectation that they would be similar. The alloy with the smallest true density, Tytin, was the only alloy significantly different from the other seven alloys, see table 5. This could be due to the higher copper content of the Tytin as compared to the others.

Since the true densities, with the exception of Tytin, are very similar, any differences in packing factor can be attributed to the differences in apparent densities. The packing factors were subjected to analysis of variance followed by comparison using Sheffe's contrast, see table 6. Possible factors that could cause the observed differences in packing factor could be alloy particle shape and/or particle size distribution.

Further investigation of these factors is indicated by the significant differences observed in the apparent densities of the eight alloys evaluated. It would also provide useful information to determine if trituration of the dry alloy powder causes a change in apparent density, especially of alloys such as Speyer, whose apparent density is very low.

The means of all the influencing factors considered for this paper are listed in table 7. The alloys are listed in ascending order based on observed leakage. The base 10 log transform values are included in the table and were used in all of the statistical analysis of the data. The same data is depicted in graphic form in figure 3. Table 8 is a matrix comprised of the correlation coefficients (r) between all possible pairs of factors.

It can be seen from the matrix of r values that no one factor stands out as being an overwhelming influence as regards leakage. The correlation coefficient between leakage and decrease in Hg following condensation was -.55. This offers little support for Granath's hypothesis. The low correlation coefficients between influencing factors is consistent with previous investigations across alloys. (2, 3, 8, 11, & 13)

The means of all the influencing factors were subjected to a stepwise-linear multiple regression analysis. One analysis was run with leakage as the

dependent variable, and condensation Hg loss, dimensional change, initial Hg, and packing factor as independent variables. Another analysis was run with dimensional change as the dependent variable, and packing factor, condensation Hg loss, and initial Hg content as the independent variables. The regression solution of both analyses were further subjected to analysis of variance. Neither regression provided a significant relationship, ($p = .05$). The analysis of variance summaries are presented in tables 9 and 10.

Conclusions

1. Plasticity, as defined by Granath, (7) cannot be used as a reliable predictor of in vitro marginal leakage.
2. Utilizing the independent factors evaluated in this study, a multiple regression analysis could not establish a significant relationship between these independent variables and either leakage or dimensional change as the dependent variable.
3. There were significant differences in the packing factors of the alloys evaluated. This infers, but does not identify, differences in physical make-up of the alloy particles.

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