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STUDIES ON MICROLEAKAGE WITH RESTORATIVE MATERIALS

III. *IN VITRO* EXPERIMENTS ON THE SEALING OF 9 BRANDS OF SILVER AMALGAM

by

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INTRODUCTION

The literature on sealing with dental amalgam shows that the results vary to some extent with the examination methods used (*Granath*, 1967). A few direct comparisons of the results obtained in leakage studies with the results of tests according to standard specifications have been performed (*Swartz & Phillips*, 1962; *Granath*, 1967). These authors found leakage with different brands of amalgam under varying conditions to agree less well with the 24-hour values for dimensional changes recorded during setting. It may be assumed that the quality of the seal depends on a number of factors such as the elasticity of the material of the mould and the adaptability of the amalgam. It was therefore thought worth while to carry out a special investigation of the influence of adaptability of the filling mixture on the seal obtainable with different brands of silver amalgam in experimental cavities in moulds whose mechanical properties were approximately the same as those of human dentin, and to place the results in relation to the values found on measurement of the dimensional changes registered with conventional testing methods.

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MATERIAL AND METHODS

The following commercially available alloys were used.

I.	DAB Argos Alloy,	Svenska Dental Instrument AB, batch no.	0601
II.	DAB Standard Alloy,	» »	4026
III.	DAB Argos non Zinc Alloy,	» »	4018
IV.	STA Amalgam-legering 68,	Guldsmeds Aktiebolaget i Stockholm, GAB, »	8640
V.	ARDENT Alloy,	Aktiebolaget Ardent, »	661001
VI.	ARDENT Alloy Extra,	» »	260504
VII.	Dental Alloy ANA 68,	Aktiebolaget Nordiska Affineriet A.N.A., »	92
VIII.	Dental Alloy ANA 68 non zinc,	» »	516
IX.	ROYAL DENTAL ALLOY medium,	Amalgambolaget AB,	not given

Alloys II and VI were coarse-cut, the others medium-cut. Moreover, II and VI were not pre-amalgamated while the others were. All except III and VIII contained zinc. As for the ratios between the alloy and the mercury, it was 5:5 for I, IV, V, VII, VIII, and IX, 5:8 for II, 5:6 for III, and 5:7 for VI.

Test bodies used for measuring dimensional changes were manufactured according to *Granath, Bladh & Edlund* (1967) with a condensing pressure of 70 kp/cm^2 and a condensing time of 30 seconds. Mixing times were selected so that they were somewhat longer than the time necessary for the alloy particles to be visually conceived as amalgamated. Three minutes after starting trituration the test body, adjusted to a length of 10.0 mm, was placed in a box with thermostatically controlled temperature. Measurements were made at 37°C $\pm$ 0.1° with a microcator (Mikrokator type 531—1, C. E. Johansson, Eskilstuna, Sweden). The measurements were obtained after another 2 minutes by photographing the dial of the microcator. Changes in length were recorded after further 10 minutes, then every 20 minutes for 3 hours, and finally after 24 hours. Details of this technique are described elsewhere (*Granath*, 1967).

The test bodies studied for leakage consisted of cylindrical moulds of dentin of the sperm whale with an outer diameter of 16 mm, an inner diameter of 5 mm, and a thickness of 5 mm. The cutting properties of dentin of the sperm whale are qualitatively comparable to those of human dentin (*Lindhe*,

1966). This presupposes, among other things, that the elastic and viscous properties of the substances are approximately equal. Alloy and mercury were the proportions prescribed by the manufacturers and mixed as in the preparation of test bodies for investigations of dimensional changes. The data are given in Table I. The amalgam was inserted according to the wet technique (*Jørgensen*, 1967), which, among other things, means that all excess mercury was removed during the actual packing process. This utilises the adaptability of the amalgam to a maximum.

Leakage was recorded with the method of *Granath & Svensson* (1970). With this method measurements are made, in a thermostatically controlled chamber, of the amount of air that passes per unit of time between the mould and the restorative material. The amalgam was packed into the moulds in the chamber where the moulds had been kept dry for a long period. The test bodies were not removed from the holder during the first 24 hours during which leakage was measured.

After all the 24-hour tests had been made, the test bodies were exposed to changes in temperature. The test bodies were first placed for 15 minutes in a refrigerator and then for a similar period in an oven. This was repeated 10 times. The temperature of the refrigerator varied between 6 and 7°C and that of the oven between 47 and 58°C. Before the final measurement of leakage, the test bodies were placed in the holder for 2 hours.

During the experimental period the temperature of the chamber and the air stream was never below 36.9°C and never above 37.1°C.

All of the tests in the entire investigation were performed as paired tests. Every amalgam test body was examined for average mercury content according to a complexometric titration procedure (*Granath et al.*, 1967).

RESULTS

The results are given in Tables I and II. The mechanically manufactured test bodies showed high precision in the paired tests as regards mercury content as well as dimensional changes. The precision of the test bodies in the paired leakage tests respecting mercury content was not so good.

Amalgam III, VII, and VIII contracted during the 24-hour period, while the others expanded. The expansion values obtained in standard tests for materials and reported by the manufacturers were nearly always higher than those given here. The leakage during the 24-hour period was substantial only for amalgam VIII and to a certain extent for amalgam III. One test body with amalgam VI showed only insignificant leakage. After changes in tem-

Table I.

Data about preparation of amalgam and dimensional changes and final mercury content of brands of amalgam used

	alloy/ mercury (g)	(≈ % Hg)	mixing time (sec.)	maximal initial contraction (μm/cm) m	d	24-hour values (μm/cm) m	d	final mercury content (%) m	d
I	1.35/1.35	(50.0)	15	− 2.75	0.5	+ 5.0	1.0	46.5	0.5
II	1.10/1.76	(61.5)	20	− 4.0	1.0	+17.0	1.0	57.5	0.5
III	1.25/1.50	(54.5)	20	−11.5	1.0	− 3.75	0.5	50.2	1.4
IV	1.35/1.35	(50.0)	15	− 1.75	0.5	+ 5.75	0.5	44.1	0.4
V	1.35/1.35	(50.0)	15	− 2.5	0	+ 3.0	0	46.3	0.1
VI	1.15/1.61	(58.4)	20	− 1.5	0	+11.0	0	55.1	0.3
VII	1.35/1.35	(50.0)	15	−10.5	0	− 5.0	0	46.8	0.3
VIII	1.35/1.35	(50.0)	20	−10.75	1.5	− 7.25	1.5	45.9	0
IX	1.35/1.35	(50.0)	20	− 3.5	0	+ 6.75	0.5	47.4	0

Registration started 5 minutes after beginning of amalgamation.
m = mean of a paired test d = difference

Table II.

Leakage) in ml air × 10^{-3} per minute at 100 mm Hg over-pressure and mercury content of test bodies*

	at maximal initial contraction	24-hour value	after change of temperature	mercury (%)
I_1	0.(2)	0.(3)	11.0	41.8
I_2	0	0	1.1	42.8
II_1	0	0	52.0	51.6
II_2	0	0	96.0	51.2
III_1	16.2	0	86.6	47.4
III_2	224.8	28.2	45.6	46.5
IV_1	0	0	10.1	37.8
IV_2	0	0	121.7	37.6
V_1	0	0	114.0	40.1
V_2	0	0	106.5	40.1
VI_1	6.3	6.9	135.3	49.3
VI_2	0	0	94.3	49.5
VII_1	0	0	113.1	40.4
VII_2	0	0	362.8	40.4
$VIII_1$	545.8	376.0	3008.0	42.2
$VIII_2$	171.3	100.2	1852.8	43.2
IX_1	0	0	122.7	42.1
IX_2	0	0	119.7	43.4

*) For practical reasons leakage smaller than 1.3×10^{-4} ml per minute was ranked as no leakage.

perature leakage was insignificant for amalgam I, extremely high for amalgam VIII, and substantial for the other types of amalgam.

The average content of mercury in the test bodies for leakage studies was invariably lower than in the mechanically condensed test bodies used for examination of dimensional changes.

DISCUSSION

Judging from the literature, it is widely believed that leakage occurs around newly inserted amalgam restorations. The present investigation produced no evidence for this assumption. This should be ascribed to the superiority of the wet technique for securing good adaptation of the amalgam to the cavity walls. The results clearly showed that the plasticity of the amalgam at the time of insertion is the factor that mainly decides the quality of the seal.

The degree of plasticity was evaluated during packing and is reflected by the difference between the original and the final mercury content. Immediately after preparation amalgams IV, V, and VII were especially plastic in view of their original amount of mercury. The least plastic, according to the same judgement, were amalgams III, VIII, and IX.

The zinc-free amalgams III and VIII were definitely less plastic than the others. The consequently poorer adaptability explains the higher 24-hour leakage with these amalgams.

All test bodies showed leakage after change of temperature. Amalgam VIII was clearly inferior to the other amalgams. The general deterioration in seal was probably due to that a greater expansion of the amalgam than of the dentin during heating caused plastic deformation of the surface profile of the amalgam as well of that of the dentin. The leakage should, however, be evaluated with caution. The dimensional changes of whale dentin as a consequence of change in temperature is dependent partly on the structure of the dentin, which may have undesirable effects on the contact relationship between the amalgam and the mould material.

The less good correlation found between dimensional changes during setting and leakage was pointed out in the introduction. This poor covariation was still more striking when the wet technique was used. This is apparent from Table II which shows that there was a substantially lower mercury content of the test bodies used in leakage studies in comparison with that of the corresponding test bodies for measuring dimensional changes. This certainly resulted in lower values for the dimensional changes of the amalgams in the leakage studies than those given in Table I. The ability of a contracting amal-

gam to give a seal preventing leakage depends probably on that an effective packing of the amalgam utilises the elasticity of the mould material. When the amalgam contracts, the elastic recovery may compensate for the effect of the contraction.

Alloys containing zinc are usually more plastic than others. Since the presence of zinc also reduces the tendency of the amalgam to corrode, the observations made in the present investigation argue for the use of zinc-containing alloys.

Summing up, the observations made appear to warrant the conclusion that high plasticity of the amalgam and proper utilisation of this property during insertion are factors of greater importance for obtaining a good seal of the cavity than is a decrease in the positive dimensional changes during setting.

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SUMMARY

The purpose was to study the effect of adaptability on the seal in a series of various brands of silver amalgam in experimental cavities in moulds whose mechanical properties were approximately the same as those of human dentin, and to place the results in relation to the values obtained for dimensional changes found with conventional testing methods.

Nine commercially available brands of alloys were included in the investigation. Two mechanically manufactured test bodies of each alloy were used for studying dimensional changes. Analysis for mercury showed the precision of the test bodies in the paired tests to be high. The precision of the test bodies concerning the dimensional changes also proved high.

The test bodies used in the leakage studies consisted of moulds made of dentin of the sperm whale with a cylindrical cavity. The cavity was filled with amalgam according to the wet technique (*Jørgensen*, 1967). Leakage was measured with the compressed air method (*Granath & Svensson*, 1970). Paired tests were made with each brand of amalgam. Analysis for mercury showed that the agreement between the test bodies in the paired tests was not quite so good as that found between mechanically manufactured test bodies.

The results showed less good agreement between the dimensional changes of the amalgams during setting and the respective degrees of leakage. It was

clearly demonstrated that it is mainly the degree of plasticity of the amalgam at the time of insertion that decides the quality of the seal. The zinc-free amalgams were much less plastic, as was reflected by the degree of leakage found.

RÉSUMÉ

ÉTUDES SUR LES DÉFAUTS MICROSCOPIQUES D'ÉTANCHÉITÉ DANS L'EMPLOI DES MATÉRIAUX POUR RESTAURATIONS DENTAIRES

III. EXPÉRIENCES IN VITRO SUR LE JOINT OBTENU AVEC 9 MARQUES D'AMALGAME D'ARGENT

Le but du présent travail a été d'étudier sur une série de différentes marques d'amalgame d'argent l'action de l'adaptibilité sur la qualité du joint dans des cavités expérimentales situées dans des moules dont les propriétés mécaniques étaient à peu près les mêmes que celles de la dentine humaine, et de comparer les résultats aux valeurs trouvées pour les changements de dimensions en employant pour les essais les méthodes classiques.

Les essais ont été réalisés sur neuf marques d'alliages existant sur le marché. Pour chaque alliage, deux éprouvettes préparées mécaniquement ont servi aux études sur les changements de dimensions. L'analyse concernant le mercure a mis en évidence un degré élevé de précision dans les deux éprouvettes utilisées. De même, un degré élevé de précision a été mis en évidence en ce qui concernait les changements de dimensions des éprouvettes.

Les essais concernant l'étude des défauts d'étanchéité ont été faits en utilisant des moules de dentine de cachalot présentant une cavité cylindrique. La cavité a été obturée à l'amalgame suivant la technique »mouillée» (*Jørgensen*, 1967). Le défaut d'étanchéité a été mesuré par la méthode de l'air comprimé (*Granath & Svenson*, 1970). Des essais doubles ont été faits pour chaque marque d'amalgame. L'analyse concernant le mercure a mis en évidence que la concordance entre les éprouvettes dans les essais doubles n'était pas aussi bonne que la concordance trouvée entre les éprouvettes préparées mécaniquement.

Les résultats ont mis en évidence une concordance moins bonne entre les changements de dimensions se produisant pendant la prise de l'amalgame et les différents degrés du défaut d'étanchéité. Il est apparu nettement que la qualité du joint dépend principalement du degré de plasticité de l'amalgame au moment de l'insertion. Les amalgames sans zinc étaient beaucoup moins plastiques, ce qui se reflétait dans leur défaut d'étanchéité.

ZUSAMMENFASSUNG

STUDIEN ÜBER MIKROLECKAGE MIT VERSCHIEDENEM FÜLLUNGSMATERIAL III. IN VITRO EXPERIMENTE ÜBER DEN VERSCHLUSS VON 9 SILBERAMALGAMSORTEN

Die Absicht war, das Adaptabilitätsvermögen verschiedener Amalgamsorten als Verschlussmaterial in experimentell geschaffenen Kavitäten bei Formen, die nahezu gleiche mechanische Eigenschaften wie das menschliche Dentin zeigten, zu untersuchen und die Ergebnisse in Relation zu den Werten von dimensionellen Veränderungen, die man mit konventionellen Testmethoden feststellen konnte, zu bringen.

Neun im Handel befindliche Amalgame wurde bei dieser Untersuchung angewandt. Man stellte zwei Testkörper von jeder Amalgamsorte auf mechanischem Wege her, um dimensionelle Veränderungen zu untersuchen. Quecksilberanalysen zeigten, dass die Präzision der Testkörper hoch war. Was die dimensionellen Veränderungen betraf, so zeigten die Testkörper ebenfalls hohe Präzision.

Die Testkörper, die zur Untersuchung von Leckage verwendet wurden, wiesen eine zylindrisch geformte Kavität auf. Dabei wurde Dentin vom Spermazetwal verwandt. Die Kavität wurde mit Amalgam nach der nassen Methode (*Jørgensen*, 1967) gefüllt. Eventuelle Leckage wurde mit Hilfe der Druckluftmethode nach *Granath & Svensson* (1970) bestimmt. Man führte zwei Teste mit jeder Amalgamsorte aus. Quecksilberanalysen zeigten, dass die beiden Testkörper nicht so gut übereinstimmten, als jene, die auf mechanischem Weg hergestellt waren.

Die Resultate zeigten, dass die dimensionellen Veränderungen des Amalgames während der Abbindung und der Grad der Leckage weniger gut übereinstimmten. Man konnte deutlich veranschaulichen, dass hauptsächlich der Plastizitätsgrad des Amalgames während des Inserierens die Qualität des Verschlusses bestimmt. Die zinkfreien Amalgame waren bedeutend weniger plastisch, wie der verschiedene Grad an Leckage wiedergab.

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