NATIONAL BUREAU OF STANDARDS REPORT

NBS PROJECT | **NBS REPORT**

0708-20-3824 | December 31, 1957 | 5757

Progress Report

INVESTIGATION OF SELECTED WAXES FOR POSSIBLE USE IN DENTISTRY

by

James E. Overberger*
Duane F. Taylor **
Gerhard M. Brauer ***
Ruth M. Davenport ****

* Guest Worker, U. S. Army, Dental Research Section, National Bureau of Standards.
** Metallurgist, Dental Research Section, National Bureau of Standards.
*** Chemist, Dental Research Section, National Bureau of Standards.
**** Physical Science Aid, Dental Research Section, National Bureau of Standards.

This work is a part of the dental research program conducted at the National Bureau of Standards in cooperation with the Council on Dental Research of the American Dental Association, the Army Dental Corps, the Air Force Dental Service, the Navy Dental Corps and the Veterans Administration.

NBS

U. S. DEPARTMENT OF COMMERCE

NATIONAL BUREAU OF STANDARDS

INVESTIGATION OF SELECTED WAXES FOR POSSIBLE USE IN DENTISTRY

Abstract

Physical properties of a number of natural and synthetic waxes were determined. The properties investigated were the coefficient of thermal expansion, flow, ash content, carvability and water solubility. The results of this study may be of value in the formulation and compounding of improved dental inlay waxes.

---

1. INTRODUCTION

One of the major sources of variation in the construction of a dental inlay is the wax pattern. Presently available inlay waxes, while capable of providing satisfactory service, have a number of undesirable characteristics. They exhibit a high coefficient of linear thermal expansion, tend to distort as a result of strain relaxation, and are subject to nonuniformity of the natural wax constituents.

Certain physical properties of a selected group of waxes were investigated to determine their suitability as bases for dental inlay waxes. These waxes included natural waxes derived from various sources and synthetics. The properties investigated were the coefficient of thermal expansion, flow, ash

content, carvability and water solubility. Although some difficulties were encountered in obtaining accurate values for the coefficient of linear thermal expansion, the precision of the measurements were of such an order that the results of this study may be helpful as a guide in the formulation and compounding of inlay waxes.

2. APPARATUS AND PROCEDURE

2.1 Coefficient of Thermal Expansion

The coefficient of linear thermal expansion was calculated from volumetric determinations by hydrostatic weighing in the range between 25° and 45°C. The procedure was adapted from that of Wiley [1]. The apparatus is shown in Figure 1. Specimen B was weighed in air and then suspended in a water bath A. The bath temperature was kept constant by a 500 watt immersion heater E, a thermoregulator D, and a stirrer C. The specimen was surrounded by an immersed polyethylene beaker having a perforated bottom. This beaker served to protect the specimen from agitation of the outer bath and, at the same time, acted as a thermal barrier to reduce the temperature variation of the specimen resulting from cycling of the heater. The temperatures in both the inner and outer baths were read to the nearest 0.1°C by means of thermometers. The maximum cycling observed in the inner bath was 0.2°C. Since the length of the cycles was small in comparison to the time required for the specimen to adjust to the bath temperature, the variation in specimen temperature is believed to have been of the same order as the precision of the

measurement, 0.1°C.

A triple Nylon filament suspended from the pan of an analytical balance was connected to a 0.25 mm nichrome wire. This wire passed through the water surface and was imbedded in the specimen. A gold sinker was attached to the end of the nichrome wire to keep the wax specimen immersed. Gold was selected as the most readily available non-corroding material having a high ratio of density to volumetric thermal coefficient. The volume of gold required to provide adequate sinking effect was so small that its thermal expansion could be ignored as having negligible effect upon the determinations. The balance employed was sensitive to 0.2 mg. as used in these tests.

Specimens of two designs were used without apparent difference in effect upon the results. The majority of the work was done employing a flat rectangular 65 x 35 x 3 mm specimen. This design was chosen in an attempt to reduce the time required for thermal equilibration of the specimen with the bath by providing large surface area and small thickness. However, it was found to be very difficult to mold non-porous specimens of this shape from certain of the higher fusing waxes. As a result, some of the determinations on the high fusing waxes, notably the polyethylenes, were made using a cylindrical speciment 60 mm long and 15 mm in diameter.

All specimens were made by gravity casting the molten wax into a mold of the proper dimensions. The flat specimens were cast in a sectional metal mold which was allowed to cool in air to room temperature. The cylindrical specimens were cast in a heated Pyrex mold and were removed by chilling below room temperature. The rate of cooling did not appear to affect the coefficient of thermal expansion, quenched and slow-cooled specimens giving similar results.

The volume of the specimen was determined first at 25°C and then at 40° to 45°C from the apparent weight in air and the apparent weight in water. The apparent weight in water at 25°C was then redetermined to see if the specimen returned to its original volume. Finally, the specimen was reweighed in air to determine any change in weight caused by solubility of the material in water. With some waxes one or more hours were required after temperature equilibrium had been established before constant weight was obtained.

Assuming the specimen to be isotropic the linear coefficient α can be calculated from the formula:

$$\alpha = \frac{V_2 - V_1}{3\,(T_2 - T_1)\,(V_1)}$$

where V_1 is the volume at 25°C, V_2 the volume at 40° or 45°C, $T_2 - T_1$ the temperature difference and 3 the factor for conversion from cubical to linear expansion to a first approximation.

A major source of error with this method is air bubbles on the specimen causing an increase in apparent volume. A few drops of Kodak Photo-Flo were added to the water to facilitate wetting of the wax and to decrease the effect of surface tension on the wire. Air bubbles adhering to the surface of the specimen were removed with a fine brush.

The sensitivity of the balance is decreased somewhat by the damping effect of the immersed specimen. Weighing errors, however, should not exceed ± 0.2 mg and the bath temperature was constant to within ± 0.2°C. Thus the uncertainty from these causes should not exceed 2% of the observed values. Determinations of the coefficients of linear thermal expansion of brass and Teflon over the range of 25° to 50°C gave results of 18 x 10^{-6}/°C and 134 x 10^{-6}/°C, respectively. These values compare with coefficients of linear expansion of 19 x 10^{-6}/°C and 138 x 10^{-6}/°C obtained by an accurate dilatometric procedure for the same specimens. Measurements of the coefficients of expansion of waxes were made at least in duplicate using a new specimen for each run.

2.2 Flow

Flow was determined by the method outlined in American Dental Association Specification No. 4 [2].

2.3 Ash Content

Ash content was determined by measuring the weight of the residue obtained after heating 1 to 2 g of the material in a crucible to 500 ± 50°C for thirty minutes in a muffle furnace and then cooling the crucible and contents in a desiccator. Results are reproducible to within ± 0.02%.

2.4 Water Solubility

Approximate water solubility was determined by weight loss between the initial weight of the sample and its weight at the end of hydrostatic weighing, usually 24 hours.

2.5 Carvability

Carvability was estimated subjectively.

3. RESULTS

Results of the measurements of the physical properties are given in Table 1.

The coefficients of linear thermal expansion over the 25° to 45°C range varied from 22 x 10^{-5}/°C for Ceroflex 1610 to 59 x 10^{-5}/°C for Baker Blue Inlay Wax. In this temperature range the three inlay waxes gave values of 52 to 59 x 10^{-5}/°C which are higher than those of the synthetic waxes or the mineral wax Ozokerite. All waxes show high coefficients of thermal expansion near their softening points. Since 45°C is near the softening points of inlay waxes, their expansion coefficients over the range from 25° to 40°C were also determined.

In this range the inlay waxes showed reduced values in their expansion coefficients but these values were still considerably higher than those of the other waxes investigated.

The thermal expansion coefficient of polyethylene waxes in the range of 25°to 45°C changes from 38 x 10^{-5}/°C to 31 x 10^{-5}/°C for samples of molecular weight of 1500 and 5000, respectively. This decrease in the thermal expansion value with increasing molecular weight occurs largely in the 1500 to 2500 molecular weight range. Similar results for polyethylenes of higher molecular weight can be found in the volume-temperature data reported by Hunter and Oakes [3].

The reproducibility of the results for AC 615, Epolene N, Ceroflex 1610 and the Petrolite PC waxes is not as satisfactory as that for the other waxes investigated. This may be caused by morphological changes during the experiments. It has been reported [3] that the density of polyethylene is dependent upon the previous thermal history of the material. Volume changes take place slowly at constant temperature as equilibrium conditions are approached. Variations in cooling rate may change the volume at equilibrium temperature. Such changes could conceivably be due to variations in the proportions of crystalline and amorphous structures or to changes in crystallite size of those waxes which undergo crystallization. X-ray diffraction studies of these waxes may help to clarify these anomalies in density behavior.

The coefficients of linear thermal expansion of the inlay waxes are considerably higher than those reported by the manufacturers. This may be due to differences in techniques of measurement. Values obtained by direct measurement of coefficient of linear thermal expansion [2] are subject to variation caused by strain relief or distortion during heating. Such strain relief may also produce distortion of the hydrostatic weighing specimens but will produce only negligible volume changes.

Water solubility of all waxes investigated is negligible, the change in dry weight is generally less than 0.04% for 24 hour immersion. Similarly, the ash content of most waxes tested is very low, being below 0.05%. The carvability of the waxes was estimated subjectively and is shown in Table 1. Only a few of the materials investigated exhibit satisfactory carvability without further modification.

Ultraflex Amber and Ozokerite 869 have flow values exceeding the limits of the American Dental Association Specification No. 4. All other waxes with the exception of the commercial inlay waxes have flow values far below those required. Thus, it may be possible to formulate materials with desirable flow and thermal expansion properties by blending of the synthetic waxes.

Because the chemical composition of most commercial waxes investigated is not known, it is not possible to correlate the

effect of composition on the properties. Additional work in this field would be very valuable.

4. CONCLUSION

Some physical properties of synthetic waxes were investigated to determine their suitability as bases for dental inlay waxes. Special emphasis was given to the determination of the coefficient of thermal expansion by hydrostatic weighing. Many of the waxes investigated have lower coefficients of thermal expansion than presently available inlay waxes. However, the softening points of those waxes which showed other desirable properties are too high to permit clinical use without addition of other ingredients.

5. REFERENCES

1. Wiley, F. Ind. Eng. Chem., 34, 1052 (1942).
2. Paffenbarger, G. C., and Sweeney, W. T. American Dental Association Specifications for Dental Materials. Specification No. 4 for Dental Inlay Casting Wax (First revision). American Dental Association, Chicago 11, Illinois. Page 25. (1956).
3. Hunter, E., and Oakes, W. G. Trans. Faraday Soc. 41, 49 (1945).

Table 1. Properties of Selected Waxes[a]

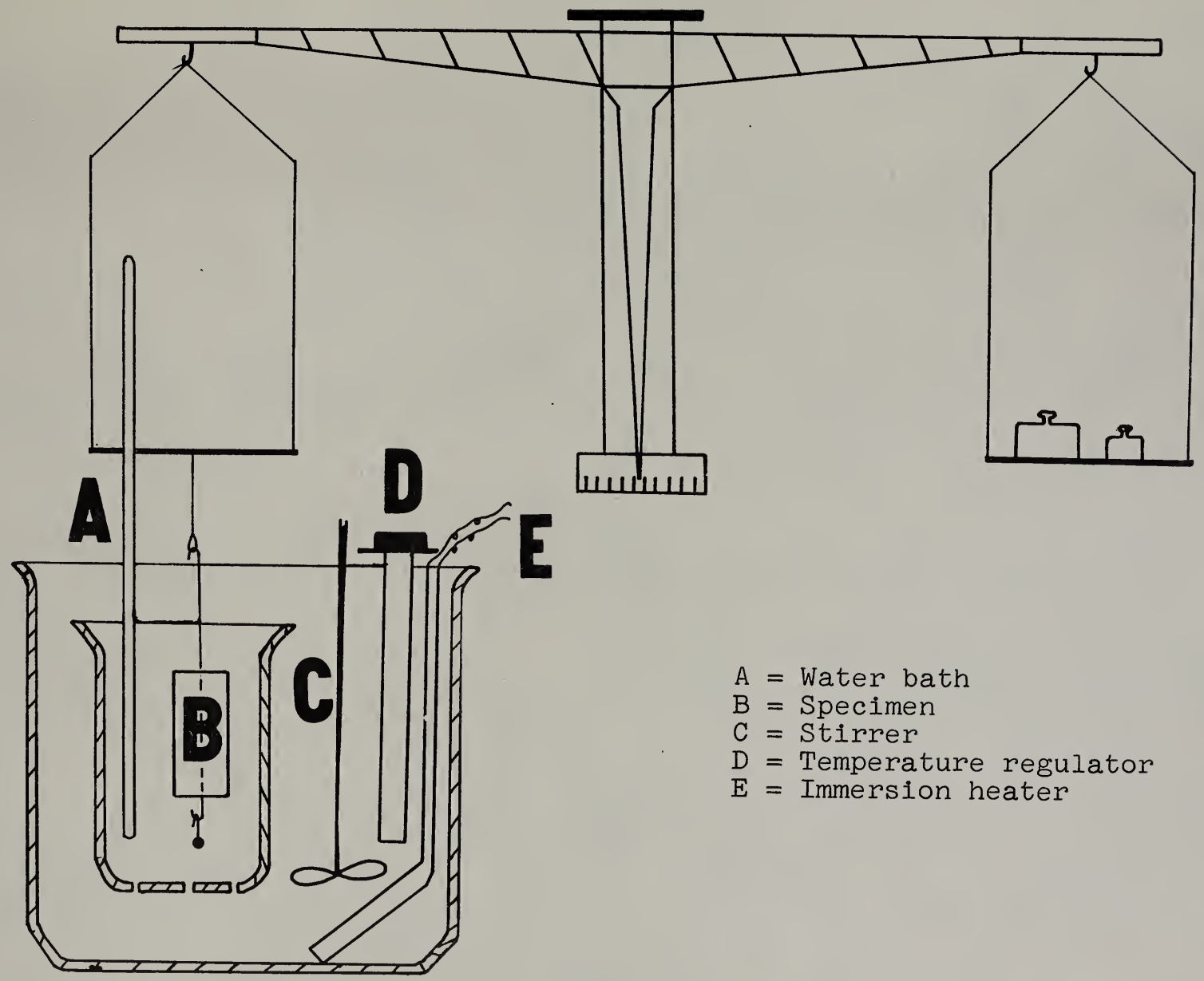
Material	Softening Point	Coefficient of Linear Thermal Expansion[b] 25°-40°C	25°-45°C	Flow 43°C	Ash Content	Carvability
	°C	x 10^{-5}	x 10^{-5}	%	%	
I Inlay Waxes						
Baker-Blue--Regular Medium	43-45	48 ± 4.1%	59 ± 1.3%	68.6	0.08	Sat.[d]
Kerr-Blue-Hard	43-45	39 ± 2.3	53 ± 1.3	59.2	trace[c]	Sat.
Taggart's Blue	43-45	42 ± 0.3	52 ± 2.3	53.2	trace	Sat.
II Synthetic Waxes						
Casting Wax 1546	69-70	-	31 ± 0.5	0.9	nil	too hard
Ceroflex 1610	101-103	20 ± 0.5	22 ± 7.3	0.4	nil	too hard
Concord #7	75	-	42 ± 0.2	-	trace	Sat.
Durawax 772	70-72	-	23 ± 1.4	1.9	0.07	too hard
Polyethylenes						
AC 617 (M.W. = 1500)	88-90	32 ± 0.9	38 ± 0.3	0.7	nil	hard
AC 6 (M.W. = 2000)	97-102	30 ± 1.4	32 ± 4.2	0.7	nil	hard
Epolene E (M.W. = 2500)	102	31 ± 2.8	32 ± 3.1	0.5	-	hard
Epolene N (M.W. = 2500)	106	29 ± 3.7	32 ± 6.5	0.0	nil	hard
AC 615 (M.W. = 5000)	102-104	29 ± 0.3	31 ± 5.6	3.5	-	hard
Poly (Vinyl Stearate)	49-51	34 ± 0.4	-	1.4	trace	too hard

a Water solubility of the waxes listed is negligible, i.e. generally less than 0.04%.
b Values shown as ± are percent coefficient of variation.
c Trace indicates values of less than 0.05%.
d Sat. = satisfactory.

Table 1. Properties of Selected Waxes[a] (Continued)

Material	Softening Point	Coefficient of Linear Thermal Expansion[b] 25°-40°C	Coefficient of Linear Thermal Expansion[b] 25°-45°C	Flow 43°C	Ash Content	Carvability
	°C	x 10-5	x 10-5	%	%	
III Mineral Waxes						
Ozokerite 869 (isoparaffin)	77-80	-	47 ± 0.2	78.2	nil	too soft
IV Petroleum Waxes						
Be Square 190/195 Amber	88-91	-	24 ± 1.2	0.1	trace[c]	too hard
Petrolite PC 13	88-91	-	45 ± 3.7	1.4	nil	hard
Petrolite PC 44	71-77	-	48 ± 4.6	-	nil	Sat.[d]
Ultraflex Amber	60-63	-	56 ± 0.2	84.5	trace	too soft

a Water solubility of the waxes listed is negligible, i.e. generally less than 0.04%.
b Values shown as ± are percent coefficient of variation.
c Trace indicates values of less than 0.05%.
d Sat. = satisfactory.



A = Water bath
B = Specimen
C = Stirrer
D = Temperature regulator
E = Immersion heater

Figure 1. Diagrammatic Sketch of Hydrostatic Weighing Apparatus