AMALGAMATION MIXTURE CONTAINING CAPSULED MERCURY
Joseph A. Bakan, Dayton, Donald D. Emrick, Kettering, and Robert C. Haines, Jr., Dayton, Ohio, assignors to The National Cash Register Company, Dayton, Ohio, a corporation of Maryland
U.S. Cl. 106—35 Int. Cl. A61K 5/02 2 Claims

ABSTRACT OF THE DISCLOSURE

A "premix" dental amalgam mixture is disclosed wherein rupturable capsules containing mercury are mixed with a predetermined amount of dental alloy such that the mixture, when triturated, forms a hardening mercury-alloy amalgam.

This is a continuation of application for U.S. Letters Pat. Ser. No. 589,846, filed Oct. 27, 1966.

This invention relates to an improvement in the formation of amalgams and compositions for the formation of amalgams in which mercury is encased in minute rigid rupturable spherical shells of polymeric material to prevent amalgamation with the other components of the complement alloy prior to the time the amalgam is to be used. More specifically, this invention relates to the formation of a "premix" of a mixture of the correct proportion of liquid mercury and alloy metal that amalgamates upon the application of stirring or pressure. An amalgam is a special type of alloy in that one of its constituents is mercury. The amalgam restoration of teeth is possible only because of the unique characteristics of mercury. It is this metal which makes possible the plastic mass which can be inserted and finished in the teeth, and which will then harden to a structure that resists the rigors of the oral environment.

Prior to this invention, mercury could not be added to an alloy prior to the time of actually filling a tooth since the resulting amalgam would harden and prevent the building of the filling, or, rather, the condensation of the amalgam.

The proportioning of the mercury and alloy is a critical factor in the success of the restoration of teeth. The amount of alloy and mercury to be used is usually defined as the alloy-mercury ratio—i.e., the parts by weight of alloy to be combined with the proper amount of mercury. For example, an alloy-mercury ratio of 5/8 indicates that 5 parts of alloy are to be used with 8 parts of mercury by weight. The ratio may vary for different alloy compositions, particle size, heat treatment and even condensation technique. The alloy-mercury ratio generally employed is 5/8, but with the use of finer grained alloys, the ratios of 5/7 or even 5/6 may be prescribed.

A wide variety of alloy and mercury dispensers, or proportioners, is available in the dental profession. They are of two general types. The most common type is the dispenser which is based on volumetric proportioning; the other type is based on measurement by weight. A considerable difference in accuracy is found among dispensers. Usually, with volumetric devices, the alloy tends to cling to the walls and corners causing incorrect proportioning.

The object of this invention is to permit the transportation of mercury in a previously proportioned "premix" or "premix" with the alloy particles for subsequent formation of an inlay filling for caries in human teeth—

the mercury in the premix being in the desired proportion to the other metals that an amalgam is formed upon the exertion of pressure, or trituration, whereby the rupturable, seamless capsule walls surrounding the mercury are ruptured and the mercury released.

This invention at once prevents the amalgamation until it is desired and allows the correct amount of mercury to be included with the alloy by encapsulating the mercury in a seamless pressure rupturable capsule which prevents amalgamation until the pressure of mulling ruptures the capsule and allows the mercury to combine with the alloy.

The present invention provides a method of packaging, storing, and handling mixtures of the accurately measured aliquots of normally liquid mercury and a solid, particulate alloy. Premature amalgamation is not possible because of the interposition of the inert capsule wall between the encapsulated liquid mercury phase and the solid fragments of the capsule wall. The mercury remains in isolation until the capsule is ruptured. In the resulting amalgam, the detritus of the ruptured wall is not incompatible with the other components of the amalgam.

The result of the instant invention is to utilize a single package of preweighed alloy pellets or powders and preweighed capsules containing liquid mercury droplets, obviating the measuring and errors inherent in prior practices. This represents a significant advance in the art over previous systems, wherein elemental mercury and the other mixed components of the amalgam were maintained in separate, isolated, relatively large containers until such time as the amalgam components were needed.

The mercury droplets can be encapsulated by any of the known methods such as that of U.S. Pat. No. 2,800,457, which issued on July 23, 1957, to B. K. Green et al. The preferred method is illustrated by the following example in which mercury droplets are encapsulated with a rigid, rupturable, seamless, wall of a gum arabic-gelatin complex.

EXAMPLE I

One hundred eighty milliliters of an 11% solution of a high quality, acid-extracted, pigskin gelatin (285—305 Bloom strength, iso-electric point pH 8—9) and one hundred eighty milliliters of an 11% solution of gum arabic in water were separately prepared. Forty milliliters of a 2% solution of a copolymer of ethylene and maleic anhydride having a molecular weight of 6000 (sold as Monsanto EMA—21 by Monsanto Chemical Corp., St. Louis, Mo., U.S.A.) and forty milliliters of a 2% solution of another copolymer of ethylene and maleic anhydride exhibiting a molecular weight ranging from 60,000 to 70,000 (sold as Monsanto EMA—31 by Monsanto Chemical Corp., St. Louis, Mo., U.S.A.) were mixed together.

The foregoing solutions were added, in sequence, to 1200 milliliters of water in a vessel, the viscosity of which had been adjusted by the addition of 4 grams of gum guar. The pH of the solution was adjusted to approximately 6.5 at 45 degrees centigrade by the addition of a 10 percent solution of sodium hydroxide after the mixture had been slowly cooled.

Approximately five pounds of pure mercury metal was dropped from a dropping funnel into the agitated mixture. The average mercury drop size was approximately 100 to 300 microns. The entire system was allowed to cool to about 25 degrees centigrade while being stirred, then chilled in an ice bath to less than 10 degrees centigrade.

To the system was added 25 milliliters of a 25%, by weight, aqueous pentaneol solution. The system was stirred for about six hours, the temperature gradually being increased to an end point of about 25 degrees centigrade, and the extraneous material decanted from the
capsules. The capsules were washed with two liters of hexylene glycol during two hours of agitation. The first hexylene glycol was decanted and the capsules were rinsed for one additional hour in an agitated two-liter quantity of hexylene glycol. The second quantity of hexylene glycol was decanted, and a vacuum Buchner funnel was used to remove the traces of hexylene glycol. Toluene was poured over the capsules and removed with vacuum filtration as a further washing step. The capsules were complete at this time.

Other examples of capsule wall materials include gelatin-gum arabic complexes, gelatin-carrageenan complexes, phase-out succinylated gelatins, and ethyl cellulose. The capsules are preferably less than 400 microns in diameter and the thickness of the capsule walls is preferably less than 10 microns thick. It must, of course, be understood as apparent that the spherical capsule diameter must be, in every case, more than twice the capsule wall thickness—such being a simple, inherent, requirement in order for the capsules to contain some material. It is obvious that a capsule cannot exist with a spherical diameter which is less than twice the capsule wall thickness.

As previously noted, the detritus of the ruptured cell walls is negligible with respect to the volume of the other reactants. The fragments do not hinder the amalgamation process in any way, nor is there any deleterious effect upon the resulting amalgam.

The component granules of the complementary alloy may, or may not, be encased in a rigid, rupturable, seamless wall. The American Dental Association Specification No. 1 requires a 65% minimum of silver, a 6% maximum of copper, a 2% maximum of zinc, and a 25% minimum of tin in an alloy for use in an amalgam alloy. In a molding test, 22.9 grams of capsules containing approximately 99% mercury internal phase, prepared in the foregoing procedure, exhibiting diameters in the range of 100 to 200 microns were thoroughly mixed under conditions of low shear and no external pressure with 14.3 grams of an alloy meeting the American Dental Association Specification No. 1 (sold as "Artisalloy" by the Baker Dental Division of Englehard Industries, Inc., East Newark, N.J., U.S.A.). The resulting mixture could be stored indefinitely in unreacted form.

A portion of the mixture was triturated with a moderate application of pressure in a glass mortar with a glass pestle for two to three minutes. The resulting amalgam exhibited a smooth consistency before it was condensed into an 8 millimeter deep 16 millimeter in diameter, cylindrical mold cavity and allowed to harden for 24 hours. The mold was removed to reveal a solid, relatively hard, durable molded specimen exhibiting no indication of weakness or faults.

As a general practice, once the amalgam is formed, it should not be permitted to stand more than 3½ minutes before its condensation into the prepared cavity. Amalgam that is more than 4 minutes old should be discarded and a new mix made.

The condensation is the forcing of the alloy particles as closely together as possible and into all parts of the prepared cavity.

The instant invention is not limited to use in preparing dental amalgams. Other uses are found in the repairing of jewelry, in soldering fine wiring, in micro-circuitry, etc.

One of the chief uses of the invention is that the encapsulated mercury does not exhibit its idiosyncratic movement and other proclivities, thereby facilitating transport and use. The encapsulated mercury is temporarily "denatured."

What is claimed is:

1. An improved dental amalgam mixture comprising a comminuted metal amalgam alloy consisting essentially of a minimum of 65% silver, a maximum of 6% copper, a maximum of 2% zinc, and a minimum of 25% tin and a plurality of droplets of mercury, each droplet of mercury being individually encapsulated in a minute, thin-walled, substantially spherical, pressure-rupturable capsule wherein the spherical diameter of the capsule is about 100 microns to about 400 microns, the thickness of the capsule wall is less than 10 microns, the capsule wall material being selected from the group consisting of copolymers of ethylene and maleic anhydride, gelatin, gum arabic, carrageenan, nucyethylated gelatin, and ethyl cellulose.

2. The dental amalgam mixture of claim 1 wherein the alloy-mercury ratio is from about % to about %, by weight.

References Cited

UNITED STATES PATENTS
1,774,258 8/1930 English 32—15
1,963,085 6/1934 Gray 32—15
2,218,755 10/1940 Kaufmann 106—35
3,016,134 1/1962 Borsuk 75—169

OTHER REFERENCES
"NCR Capsules Have Wide Possibilities," an article appearing in the October 1959 issue of The NCR Factory News, the National Cash Register Company, Dayton, Ohio.

JULIUS FROME, Primary Examiner
J. H. MILLER, Assistant Examiner

U.S. Cl. X.R.
32—15; 75—169; 106—125, 129, 163; 252—316; 260—875