

UNITED STATES PATENT OFFICE

2,397,145

IMPRESSION MATERIAL

Homer van Beuren Joy, Montclair, N. J., and
Eugene J. Molnar, Toledo, OhioNo Drawing. Application December 11, 1943,
Serial No. 513,994

9 Claims. (Cl. 18-47)

This invention relates generally to impression-taking materials containing water-soluble alginates and particularly to dental impression materials.

The object of the invention is to provide an impression material which, when mixed with water and applied to a structure the reproduction of which is desired, sets in a controllably short time to yield a stiff elastic gel. Another object is to obtain a material which can be used to take an impression of parts of the human mouth without discomfort to the patient. Since a defect of many impression materials based on water-soluble organic reversible gel-forming colloids is their short shelf-life (due in most cases to absorption of water on storage and the resulting premature reaction), an object is to provide a non-hygroscopic material which is stable on storage. Also, since many impression materials after setting to shape have an adverse effect on the setting of plaster when a cast of the impression is made, it is an object to prepare a material which when set can be used directly in contact with plaster to make a model.

It is desirable that a material which is to be used for taking impressions should be easily brought into condition for use; it should require no heat to bring it to proper working consistency or to cause its setting; it should be of such physical condition that it can be inserted into cavities and fissures and readily removed therefrom without destruction when set; it should set in a suitable time which is not so short as to prevent proper manipulation in application nor so long as to cause discomfort to a dental patient; it should form a sharp accurate impression of the part to which it is applied; it should set to a tough rubbery state so that it can be stripped from the structure to which it has been applied even when undercuts are present; the dry powder composition should be stable over a long period before mixing with water for use; and finally the impression or negative should be capable of being used directly for a positive plaster cast or model.

We have found that impression materials which meet the above requirements can be made from water-soluble alginates and certain alkaline-earth chromates. A filler is preferably added and also, where desired, a retarding or accelerating catalyst to regulate the setting time. Advantages of the chromate-containing materials are that, when mixed with water, the time interval between the plastic stage and full set is short, the impression need not be treated with a "tanning

solution" before pouring the plaster model as is the case with such compositions as those containing calcium sulfate and alginate, and the mouth is left in a cleaner state; i. e., with fewer residual loose particles. Strength, toughness and hardness are highly satisfactory.

The alkaline-earth chromates which have proper solubility and which we have found to be valuable setting agents for alginates are those of calcium and strontium. These may be obtained by several methods including wet or dry procedures. Purity varies with different methods of preparation and likewise the speed of setting when they are mixed with the alginate. Thus, pure strontium chromate acts more slowly than calcium chromate. However, we have found that by adding a suitable accelerator (preferably a small amount of ammonium chromate) an excellent dental material may be obtained from strontium chromate.

The chromate-containing compositions are very sensitive to variations in formulation. If a given formula is not properly balanced it does not give the best results. To influence the setting time we may change the proportion of water (decreasing the water makes it set faster) or the temperature of the water. Also, to produce faster setting we may add various accelerators, such as ammonium chloride, ammonium benzoate, ammonium sulfate, zinc sulfate, and the like. For example, ammonium chloride, when added to calcium chromate in an amount as low as 1%, hastens setting without changing the properties of the set material. The pH seems to be an important variable. For example, a certain strontium chromate material gave a better result when a small amount of disodium phosphate was added than with trisodium phosphate. Also, the degree to which the chromates are dried influences the speed of setting. A highly hydrated calcium chromate is much more rapidly soluble in water than when partially dehydrated and consequently the setting time may be too fast and must be retarded by the addition of a small amount of a salt which precipitates calcium or strontium such as a water-soluble phosphate, oxalate, carbonate and the like. Likewise, a partially dehydrated salt is more stable on exposure to air and is less water-absorbing.

Although calcium and strontium chromates from any source can be used to convert aqueous alginate compositions to tough elastic gels, we give below preparations which are of preferred degree of activity.

(A) A solution of 105 parts technical sodium

chromate in 80 parts water is added to a solution of 85 parts calcium chloride in 160 parts water. The precipitate is filtered, washed and dried for about 15 hours at 200° F. The dry material is sieved to about 100-mesh and further dried for 5 hours at about 200° F.

(B) A solution of 120 parts ammonium chromate in 100 parts water is added to a solution of 150 parts calcium chloride in 320 parts water. Filter, wash and dry as in A.

(C) Add 40 parts powdered anhydrous calcium chloride to a solution containing 40 parts technical chromic acid anhydride, 70 parts 28% ammonium hydroxide and 150 parts water. The precipitate is filtered, washed and dried at about 200° F.

(D) To a solution containing 40 parts technical chromium trioxide and 70 parts 28% ammonium hydroxide in 150 parts water is added with stirring 40 parts powdered strontium chloride. The precipitate is filtered, washed and dried.

The alginates used herein include potassium, ammonium and preferably sodium alginates. As fillers we use any substantially water-insoluble powder such as magnesium carbonate (light or heavy), calcium carbonate, mica, stearates, hard water-insoluble waxes, clays, silica, barytes, magnesium oxide, magnesium phosphate (tribasic), zinc oxide and the like. The type and amount of filler has an importance in regard to the qualities of the final product; its smoothness, mixing qualities and setting time.

The powdered alginate is intimately mixed with the finely divided chromate and filler, together with any other materials such as accelerators or retarders (where desired) or pigments, dyes, flavoring or perfume materials and the like. Immediately before use the powder is mixed with the requisite amount of water to form a smooth paste and applied to the part (mouth or other body part, industrial or artistic figures, etc.) which is to be reproduced. After the composition has set, it is stripped as a tough elastic gel which can be used as a mold for the casting of a model.

The following are illustrative examples of impression materials and are not to be considered as limiting since the proportions can be changed to a certain extent to meet individual requirements. Parts are by weight.

Example 1

	Parts
Calcium chromate (Preparation A)-----	1
Sodium alginate-----	1
Zinc oxide-----	1
Light magnesium carbonate-----	4

This powder is mixed with water (1 part powder to 4 parts water). Used as a dental impression material, the powder is added to the water and spatulated for 1½ minutes, then placed in the tray and inserted into the patient's mouth. In 3 minutes the composition has set to a stiff gel and can be removed. After rinsing in water, plaster is poured into the impression and after hardening the gel is stripped, leaving an accurate plaster model.

Example 2

	Parts
Sodium alginate-----	3
Calcium chromate (Baker's C. P. powder, basic)-----	3
Calcium carbonate-----	10
Powdered mica-----	5
(Mix with 84 parts water.)	75

Example 3

	Parts
Potassium alginate-----	1
Calcium chromate-----	1
Heavy magnesium carbonate-----	4
Light magnesium oxide-----	1
(Mix with 28 parts water.)	

Example 4

	Parts
Sodium alginate-----	6
Strontium chromate (Preparation D)-----	6
Trisodium phosphate-----	1
Powdered hard wax such as carnauba-----	4
Zinc oxide-----	6
Magnesium carbonate-----	20
(Mix with 172 parts water.)	

Example 5

	Parts
Sodium alginate-----	1.5
Strontium chromate-----	1.2
Ammonium chromate-----	0.25
Disodium phosphate-----	0.6
Magnesium carbonate-----	12
(Mix with 50 parts water.)	

Example 6

	Parts
Potassium alginate-----	1.7
Strontium chromate-----	1.7
Ammonium carbonate-----	0.5
Precipitated chalk-----	16.0
(Mix with 50 parts water.)	

Examples 2-6 are used in the same way as Example 1.

We claim:

1. An impression material comprising a water-soluble alginate, a filler and an alkaline-earth chromate hardening agent selected from the group consisting of calcium and strontium chromates.
2. An impression material comprising a water-soluble alginate, a filler and calcium chromate.
3. An impression material comprising sodium alginate, a filler and calcium chromate.
4. An impression material comprising a water-soluble alginate, a filler and strontium chromate.
5. An impression material comprising sodium alginate, a filler and strontium chromate.
6. An impression material comprising a water-soluble alginate, a filler and an alkaline-earth chromate hardening agent selected from the group consisting of calcium and strontium chromates, the alginate and chromate being in substantially equal parts by weight.
7. An impression material comprising potassium alginate, a filler, and an alkaline-earth chromate hardening agent selected from the group consisting of calcium and strontium chromates.
8. An impression material comprising a water-soluble alginate, a filler and an alkaline-earth chromate hardening agent selected from the group consisting of calcium and strontium chromates, and an accelerator.
9. An impression material comprising a water-soluble alginate, a filler and an alkaline-earth chromate hardening agent selected from the group consisting of calcium and strontium chromates, and a retarding agent.

HOMER VAN BEUREN JOY.
EUGENE J. MOLNAR.