

UNITED STATES PATENT OFFICE

2,465,773

PROCESS OF PRODUCING A CATALYTIC ARTICLE

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No Drawing. Application February 21, 1947,
Serial No. 730,209

2 Claims. (Cl. 204—24)

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This invention relates to the production of a new and improved catalyst, and more particularly it relates to a catalytic structure comprising a lead oxide.

It is an object of this invention to produce a solid catalyst structure comprising a lead oxide which is particularly suitable for use in the decomposition of hydrogen peroxide.

It is another object of this invention to produce a solid catalyst structure comprising a lead oxide which is particularly suitable for use in the decomposition of hydrogen peroxide and which has a large surface area.

Other objects of the invention will appear hereinafter.

The objects of this invention are accomplished by forming an adherent coating of anodically oxidized lead on a metallic supporting structure. This may, in general, be accomplished by coating a metallic supporting structure, for example, a supporting structure of iron, steel, copper, brass, bronze, nickel, or the like, with lead, for example, by dipping the structure in molten lead, by adhering lead sheets or films to the structure with a bonding composition, or by electrodepositing lead on the structure, and anodically oxidizing at least the external surface of the lead coating in an alkaline electrolytic solution. The anodic oxidation is preferably carried out in such a manner as to obtain a rough coating of large surface area.

The anodic oxidation may be produced by placing the lead coated structure as the anode in an alkaline electrolytic solution containing a steel cathode and passing an electric current therethrough.

In accordance with the preferred procedure a formless iron or steel structure, for example, a meshed screen or a perforated sheet of iron or steel is provided with a coating of lead by electro-deposition from a lead electroplating bath. The electro-deposited coating of lead is then subjected to the action of an aqueous solution of an alkali metal (sodium or potassium) cyanide with the lead-coated structure constituting the anode of an electric current passing through said solution, and then subjecting the structure to the action of an aqueous solution of an alkali metal (sodium or potassium) carbonate with said structure constituting the anode of an electric current passing through said carbonate solution.

The iron or steel supporting structure for the solid catalyst of this invention preferably consists of commonly-known iron or steel wire

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screen having a mesh size between $\frac{1}{8}$ inch and $\frac{1}{4}$ inch. Such iron or steel screen may be obtained as plain screen without a protective coating or as an unlacquered-galvanized or lacquered-galvanized iron or steel screen. When the screen contains no protective coating or when it contains a lacquer coating, it is preferably first cleaned with an alkaline cleaning solution, for example, an aqueous solution containing 8 to 16 oz. per gallon of caustic soda. The screen may be cleaned in such solution by immersing the same in the solution having a temperature of 90° C. to 100° C. for a period of ten minutes and then spray-rinsing the same with water. Other alkaline cleaning solutions commonly used in the arts can be used as well.

The screen, whether or not it is previously cleaned in an alkaline bath, is preferably subjected to acid pickling, for example, in an aqueous solution of sulfuric acid containing 3% to 5% acid and having a temperature around 20° C. to 30° C. The screen may be immersed in the acid pickling bath for a period of three to five minutes or as much longer as necessary to remove the galvanizing (zinc) therefrom and produce a clean steel surface. The acid pickling can be carried out in any other known acid pickling solution which will produce a clean steel surface.

The clean steel screen may next be subjected to the lead plating operation. Although not essential, it is preferred to first provide the screen with a light coating of copper commonly referred to as a copper strike. This may be done conveniently, for example, by positioning the screen as the cathode together with a steel anode in a bath composed of 2 to 6 oz. per gallon of copper cyanide, 4 to 8 oz. per gal. sodium cyanide (alternatively 5.5 to 8.0 oz. per gal. potassium cyanide), said bath having a temperature of 30° C. to 70° C., and using a current density of 15 to 45 amperes per sq. ft. at an electromotive force of 4 to 6 volts for a period of 3 to 6 minutes.

The screen, with or without the copper strike, is then plated with lead. This is preferably done by an alkaline lead-electroplating process. A process such as disclosed in the abandoned co-pending application of Cliver and Wernlund, Serial No. 507,025, may be used to good advantage for this purpose. A suitable lead plating solution, for example, may contain 8 to 12 oz. per gal. of lead sulfocyanide, 12 to 20 oz. per gal. of caustic soda, 6 to 8 oz. per gal. of glycerine, 1 to 3 oz. per gal. of trimethyl-C-cetyl alpha-betaine. This lead plating bath may be maintained at a temperature

of 75° C. to 100° C. As anodes, the bath should preferably contain substantially pure lead bars or sheets. The screen is positioned in the bath as the cathode. As anode current density between 10 and 20 amperes per sq. ft., a cathode current density of between 10 and 25 amperes per sq. ft., and an E. M. F. between 0.3 to 0.6 volt are preferably employed. A lead plate of 0.002 to 0.01 inch is sufficient for the purposes of the present invention. A coating of about 0.006 inch in thickness, which is very satisfactory, will be obtained in a plating period of about 2 hours and 50 minutes employing a current density of about 20 amperes per square foot at both electrodes.

After the lead plating operation, the lead plating is "anodized" by positioning the plated structure as the anode in an aqueous solution containing 3 to 5 oz. per gal. of sodium cyanide (or 5 to 6 oz. per gal. of potassium cyanide) and having a temperature of 20° C. to 30° C. Sheet steel cathodes are positioned in the bath and an electric current having a current density of 8 to 12 amperes per square foot and an E. M. F. of 4 to 6 volts is passed through the bath for about 10 minutes. This anodic treatment apparently produces an adherent film of lead cyanide on the lead plating.

The lead cyanide film on the structure is then converted to a lead oxide by a second anodizing treatment in an aqueous solution containing 10 to 15 oz. per gal. of potassium carbonate (or 8 to 12 oz. per gal. of sodium carbonate) and having a temperature of between 20° C. and 30° C. The said lead cyanide coated structure is made the anode and with sheet steel cathodes a current of 10 to 20 amperes per square foot at an E. M. F. of 4 to 6 volts is passed through the bath for one to two hours. At 10 a./s. f. a 90 minute anodization will produce a very suitable lead oxide coating. The lead oxide coating may vary in color from green to red and therefore may consist of a mixture of different lead oxides or the particular lead oxide obtained may vary depending upon current density, length of anodization time, purity of carbonate, or the like.

As a result of the above-described anodization treatments, the coated structure will be rough and uneven having the approximate appearance and feel of rough sand paper. When a lead-electroplated iron screen is anodized in a 3% aqueous sodium cyanide solution having a temperature of 25° C. for 10 minutes at 20 amperes per square foot and is then anodized in a 10% aqueous solution of sodium carbonate having a temperature of 25° C. for 90 minutes at 10 amperes per square foot, the lead oxide film will comprise approximately 14 grams per square foot of screen.

Although it is not essential, it is preferred to rinse the screen structures between all cleaning, plating, and anodizing treatments to avoid contaminating succeeding baths.

A useful catalyst for the decomposition of hydrogen peroxide can be obtained by omitting the step of anodizing in the alkaline cyanide and directly anodizing the lead-coated structure in an alkali metal carbonate solution as above described.

The catalytic structures produced in accordance with the present invention are particularly useful as decomposition catalysts for hydrogen peroxide solutions containing from 27% to 100% H₂O₂ by weight. They may, however, be used

wherever anodically oxidized lead has a useful function as a catalyst. When used as decomposition catalysts for hydrogen peroxide, it is only necessary to immerse the catalytic structure in an aqueous solution of hydrogen peroxide.

Reference in the specification and claims to parts, proportions and percentages, unless otherwise specified, refers to parts, proportions and percentages by weight.

Since it is obvious that many changes and modifications can be made in the above-described details without departing from the nature and spirit of the invention, it is to be understood that the invention is not to be limited to said details except as set forth in the appended claims.

What is claimed is:

1. The process of producing a catalytic article which comprises electrodepositing a lead coating on a foraminous iron supporting structure, subjecting said lead-coated structure for a period of about 10 minutes to the action of an aqueous solution containing 3 to 6 oz. per gal. of an alkali metal cyanide with the structure constituting the anode of an electric current having a current density of 8 to 12 amperes per sq. ft. and an E. M. F. of 4 to 6 volts passing through said solution, and then subjecting said structure for a period of 1 to 2 hours to the action of an aqueous solution containing 8 to 15 oz. per gal. of an alkali metal carbonate with the structure constituting the anode of an electric current having a current density of 10 to 20 amperes per sq. ft. and an E. M. F. of 4 to 6 volts passing through said carbonate solution.

2. The process of producing a catalytic article which comprises electrodepositing a lead coating on a foraminous iron supporting structure, subjecting said lead-coated article for a period of about 10 minutes to the action of an aqueous solution containing 3 to 5 oz. per gal. of a sodium cyanide with the lead-coated structure constituting the anode of an electric current having a current density of 8 to 12 amperes per sq. ft. and an E. M. F. of 4 to 6 volts passing through said solution, and then subjecting the structure for a period of 1 to 2 hours to the action of an aqueous solution containing 8 to 12 oz. per gal. of sodium carbonate with said structure constituting the anode of an electric current having a current density of 10 to 20 amperes per sq. ft. and an E. M. F. of 4 to 6 volts passing through said solution.

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