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## PROCESS FOR THE PRODUCTION OF ACTIVE METALLIC CATALYSTS.

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*To all whom it may concern:*

Be it known that I, ERNEST JOSEPH LUSH, a subject of the King of Great Britain, and resident of London, England, have invented a Process for the Production of Active Metallic Catalysts, of which the following is a specification.

In the use of metallic catalysts, and more particularly in the use of nickel in the hydrogenation of oils, various forms of metal have been proposed such as wire turnings, plates, ribbons, wool and various methods of rendering the surface more active have been suggested. For example, it has been proposed to tarnish such surfaces by oxidation with air, oxygen or ozone or roughen such surfaces by the electrolytic deposition of metal. As far as I am aware the results of such activation are not such as to produce a catalyst comparable in activity to that produced by the use of precipitated metallic salts reduced on supports in hydrogen, as, for example, nickel carbonate precipitated on kieselguhr and reduced in hydrogen.

I have discovered a method of activating metallic surfaces and restoring such activity in spent catalysts as will enable these forms of nickel to more than compete with precipitated catalysts above referred to. Moreover such highly active surfaces permit of the more economical use of catalysts since with increased activity such as I am able to produce full use can be made of such additional advantages as follow from the use of stationary catalytic material, as for example, the continuous passage of the material to be treated through the catalytic mass. Moreover such highly active material as is produced by my process enables continuous working to be carried on for longer periods than hitherto possible.

According to my invention nickel in any convenient form, such as wool, turnings, plates, ribbons, rods, or balls for example or other metal is made the anode in an electrolytic bath using as electrolyte a salt which will not allow nickel or other metal to pass into solution, with the result that on passage of the electric current an active precipitate is coated over the metal which on reduction in hydrogen, at the lowest temperature at which the metal can be formed, results in the production of a very active form of catalyst.

I have further found as for example in the use of nickel prepared in this way for the hydrogenation of oils that the above described method of activating metallic surfaces can be successfully applied to spent catalysts. After removal of the oil by extraction with a solvent, preceded where desirable by centrifugal separation of adhering oil, or more simply by roasting, this treatment results in the complete removal of the poisons which may be for example carbon monoxide removed as carbon dioxide or sulphur compounds usually present in the catalyst as metallic sulphides removed as sulphates. By this means I have found the original activity of the catalysts is completely restored and in some cases actually considerably increased. This revivification can be repeated indefinitely.

As an example of the working of my invention the production of a catalyst suitable for the hydrogenation of oils may be effected in the following manner:—

Pure soft nickel rod is turned on a lathe and the turnings packed in asbestos cloth are made the anode in an electrolytic bath containing as electrolyte a 1% to 5% solution of sodium carbonate. A sheet of nickel is used as cathode and a current of suitable density with a carefully controlled voltage (3–4 volts) is passed through until the anode surface is blackened uniformly throughout.

To produce the oxidation of the metallic surface within a given time the amperage must be varied according to the nature of the metal to be treated and the period within which the oxidation takes place. As an example I have found that for one pound of fine nickel turnings a current of one ampere hour at 3 to 4 volts produces a satisfactory catalyst.

Agitation by air or by mechanical means such as a revolving anode may be used to promote the speed and uniformity of the oxidation of the surface. The anode turnings are then removed from the bath, washed with water to free from electrolyte, dried and reduced in hydrogen at 250° C. for a comparatively short time, for example one half to one hour when an active catalyst will be obtained suitable for hydrogenation of oils or other substances.

As an example of the use of such a catalyst it has been found that previously puri-

fied oil can be run through these reduced activated turnings contained in a suitable vessel charged with hydrogen for several weeks, the speed of flow controlling the degree of hydrogenation and the hydrogenated oil leaving the vessel clear and bright without detaching the active surface. In this way the process of hydrogenation of oil may be more economically effected than has hitherto been possible due to the uniform production of a very active surface on the metal.

In the preparation of a catalyst as above described the electric current is responsible for a certain amount of disintegration of the nickel and in order to avoid waste of the same this may be removed from the bath and recovered by electrolytic deposition upon rods or bars from whence it may be turned or otherwise used.

When it is desired to produce a catalyst of the known powder form a modification of the above described process may be employed whereby the nickel anode may be rapidly converted into a very finely divided precipitated salt of nickel, e. g., hydrated nickel carbonate, by variations in the conditions of the electrolytic bath.

For example, by the use of a sufficiently high voltage or by mixed salts in the electrolytic bath (one of which forms a soluble, the other an insoluble salt of nickel—as, for instance, sodium nitrate and sodium carbonate) the nickel anode may be quantitatively transformed to hydrated nickel carbonate. This precipitate being formed from dilute solutions and (where necessary) in an electrolyte consisting of water with only enough sodium carbonate in solution to raise the conductivity sufficiently, results in a precipitate more free from adsorbed salts than that prepared by the usual mutual precipitation methods hitherto employed, e. g., precipitation of nickel sulphate with sodium carbonate.

The precipitate thus formed is then removed from the bath, washed with water to free from electrolyte dried and reduced in hydrogen at 250° C. for a comparatively short time for example one-half to one hour resulting in a powder catalyst suitable for the hydrogenation of oils or other substances.

What I claim is:—

1. A process for the production of active metallic catalysts which consists in subjecting the metallic surfaces to an electrolytical anodic oxidation and afterwards reducing in hydrogen substantially as described.

2. A process for activating nickel surfaces for catalytic purposes which consists in subjecting the mass to an electrolytical anodic oxidation in an electrolytic bath containing salts of an alkali as electrolyte which will not allow the nickel to pass into solution and afterwards reducing in hydrogen.

3. A process for the production of a powder nickel catalyst which consists in subjecting the metallic surfaces within an electrolytic bath to a continued electrolytical anodic oxidation so that the said nickel is rapidly converted in a very finely divided precipitated salt of nickel said precipitate being subsequently removed from the bath washed with water dried and reduced in hydrogen at 250° C.

In testimony whereof I affix my signature.

ERNEST JOSEPH LUSH.