E. C. KAYSER.

METHOD OF SATURATING FATTY ACIDS OR THEIR GLYCERIDS WITH HYDROGEN.

APPLICATION FILED FEB. 18, 1910.

1,008,474. Patented Nov. 14, 1911.

Fig. 1.

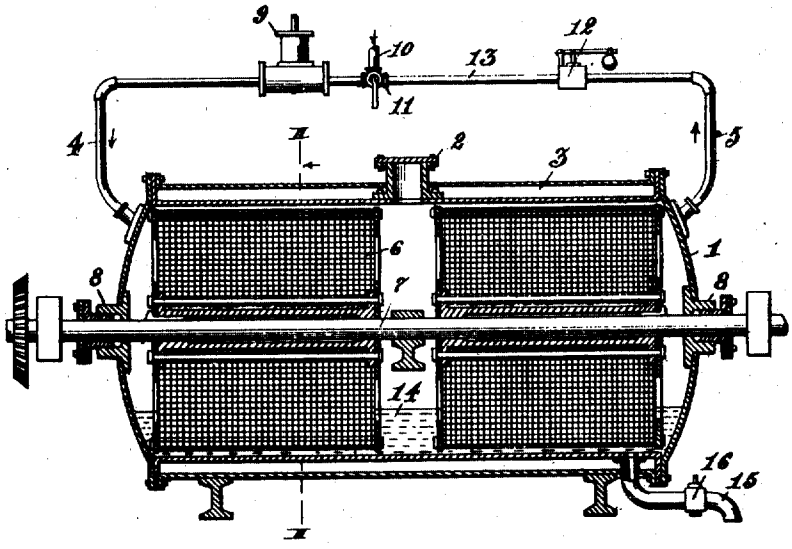
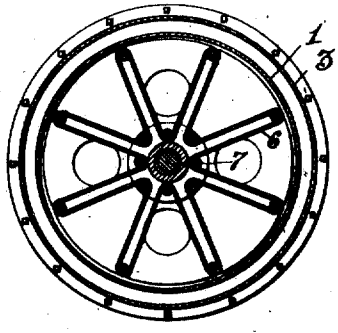


Fig. 2.



Witnesses:
N. P. Leonard.
C. H. Potter.

Inventor
Edwin Burns Kayser,
by Byrnes, Townsend & Brockett,
Attys.

UNITED STATES PATENT OFFICE.

EDWIN CUNO KAYSER, OF CINCINNATI, OHIO, ASSIGNOR TO THE PROCTOR AND GAMBLE COMPANY, OF CINCINNATI, OHIO, A CORPORATION OF OHIO.

METHOD OF SATURATING FATTY ACIDS OR THEIR GLYCERIDS WITH HYDROGEN.

1,008,474. Specification of Letters Patent. Patented Nov. 14, 1911.

Application filed February 18, 1910. Serial No. 544,630.

To all whom it may concern:

Be it known that I, EDWIN CUNO KAYSER, a subject of the King of Great Britain, and a resident of the city of Cincinnati, in the county of Hamilton and State of Ohio, United States of America, have invented certain new and useful Improvements in Methods for Saturating Fatty Acids or Their Glycerids with Hydrogen, of which the following is a specification.

It is by now well known that unsaturated fatty acids, their glycerids or other esters, in the fluid state, can be made to absorb gaseous hydrogen, until they become converted into the corresponding semi-saturated or saturated compounds, by the intervention of certain finely divided metals, nickel in particular. For that purpose, the nickel-powder is kept in suspension in the hot oil or fatty acid, while hydrogen gas is passed through, or otherwise brought in suitable contact with, the mixture.

In order to sufficiently expedite the transfer of hydrogen for practical purposes, considerable quantities of nickel have to be present. This metal must take the form of an almost impalpable powder and in consequence, when it comes to its separation from the product of reaction, it will be found to form an almost impenetrable sediment on the filter cloth, a fact greatly interfering with the technical applicability of the process. Moreover, for reasons but imperfectly understood, but apparently due to the deposition thereon of carbonaceous by-products, such pure metal soon loses its catalytic activity and cannot be made to repeatedly answer its purpose.

In the catalytic production of sulfuric acid, and in similar industrial processes, where reaction between gases is to be induced, it is customary to spread the catalytic metal or other catalyzer over fibrous materials like asbestos, over fragments of brick or the like inert materials. Since the "catalyst" in these cases remains stationary, such measures are obviously necessary in order to facilitate the transverse movement of the gas through the catalyzer, or in order to produce more contact than a mere passage over a pulverous layer of metal would afford. In the present instance, however, when the metallic particles are already kept apart and readily accessible by their suspension in a liquid medium, it would be considered unnecessary to disseminate such metal over an inert vehicle and the presence of the latter would be judged more likely to impede than to facilitate the reaction. I have, notwithstanding this, ascertained, however, that nickel firmly and evenly deposited upon a voluminous, pulverous inert material exercises in a fluid medium a far more energetic action, and much longer retains its vitality than an equal weight of pure metal in the finest state of division procurable. The method of thus depositing on or coating the pulverous inert material with the nickel powder forms no part of my present invention, which relates to the use of such pulverous nickelized material in the saturation of oils and fats with hydrogen. It will, therefore, be sufficient to state that in order to prepare my catalytic agent, I bring suitable soluble or insoluble compounds of nickel in an appropriate manner into intimate combination with an inert absorptive and comparatively bulky mineral substance such as kieselguhr. I then dry and comminute the product, and reducing the powder thus produced with hydrogen, a highly efficient and reliable nickelized, inert pulverous material is the result.

In order to carry out my process, I introduce into a suitable closed receptacle a quantity of the mixture of my fatty material and the pulverized, nickelized inert support, and by means of paddles or stirrers, I thoroughly agitate the mixture while introducing the hydrogen. The vessel may either be filled with the charge, in which case the gas has to be atomized within the fluid, or the vessel may be only partly filled, so as to permit the agitators or paddles to propel or project the charge into a surrounding atmosphere of hydrogen. By preference, the hydrogen is supplied under pressure, but a certain amount is always allowed to continuously escape in order to carry off generated steam and other gaseous by-products.

Under suitable precautions, water gas or other gas mixture sufficiently rich in hydrogen gas may be used in place of pure hydrogen. When pure hydrogen is used, under artificial pressure, the gas outlet should be closed by a weighted valve, and the gas supply so regulated as to constantly maintain the desired pressure.

The temperature of the oil mixture may vary within considerable limits. A temperature from one hundred and fifty degrees centigrade to one hundred and sixty degrees centigrade will, as a rule, be found desirable.

The great technical advantage secured by my process in which the nickel powder is firmly and evenly distributed upon the voluminous, pulverous inert material, when mixed with the oil or fat to be treated, may in part be attributed to the ultra-division of the metal, attainable by suitable chemical incorporation, with the inert vehicle, wherein it approaches the colloidal state. It may, however, with equal force be ascribed to a direct coöperation between metal and "inert" body, the latter, present in preponderating quantity and volume, absorbing the hydrogen in the first instance and then yielding it to the metal, which in turn transmits it to the fatty body. The prolonged vitality of the catalyzer may on the other hand be due either to a modified action of the disseminated metal and to a diminished formation of by-products, or to the retention thereof by the inert vehicle, or finally to the more constant physical structure of the metallic particles. Whatever be the cause of the increased activity and prolonged vitality of the metalliferous pulverous catalyzer over the pure metal, the direct technical advantage attained by the change is sufficiently conspicuous. This advantage is further advanced by two other factors, greatly pronounced when using a vehicle like kieselguhr, namely the comparatively low specific gravity and the easy permeability of the catalyzer. The former greatly facilitates suspension in the surrounding fluid, the latter renders rapid and neat separation from the fatty substance by the conventional use of the filter-press an easy matter. Finally, the inert material, which forms by far the principal constituent of a well-made catalyzer, exercises a decolorizing and deodorizing action upon the material treated, far beyond that observed with an equal proportion of pure metal.

Suitable apparatus for carrying out the process is shown in the accompanying drawing, in which—

Figure 1 is a vertical axial section; and Fig. 2 is a vertical transverse section on the line II—II of Fig. 1.

The apparatus illustrated comprises a stationary horizontal cylinder 1, having a manhole 2 for the introduction of oil and catalyzer, a steam jacket 3, hydrogen supply- and discharge-pipes 4, 5, and revolving wire-gauze beaters or agitators 6, carried by a central shaft 7 journaled in stuffing-boxes 8 in the ends of the cylinder. Hydrogen is supplied to the compression-pump 9 through a pipe 10 having a three-way or other cock 11. The unabsorbed excess of hydrogen escaping from the drum through the pipe 5 passes a weighted relief-valve 12 and is thence returned by a pipe 13 to the pump. The liquid mixture of oil and catalyzer to be treated in the cylinder is indicated by the line 14. The product is delivered through the tap 15 having the cock 16.

In practice, the oil to be hydrogenized, for example cotton-seed oil, is preferably charged into the cylinder through the manhole 2 to about the level indicated. A relatively small amount of the nickelized catalyzer, usually less than one per cent. by weight of the oil, is also introduced. The mixture is then heated by steam in the jacket 3 to a temperature of about 150° to 160° C. Hydrogen is pumped into the cylinder through the pipe 4, the unabsorbed excess escaping through the pipe 5 and relief-valve 12, and the wire-gauze beaters are set in motion. The heat, supply of hydrogen and agitation are continued until the oil has been hydrogenized to the desired degree, as shown by its consistency or by determination of its titre and iodin value, usually for several hours and until it has been converted into a product which is solid or semi-solid when cold. The cock 16 is then opened, the mixture of hydrogenized oil and catalyzer is run out through the tap 15, and the catalyzer is filtered out for re-use.

What I claim as new, and of my invention and desire to secure by Letters Patent, is:—

1. The process of hydrogenizing fatty acids and their esters, which consists in agitating in the presence of hydrogen a heated mixture of the fatty acid or ester and a pulverulent catalyzer consisting of a light, voluminous, finely-divided, inert support impregnated with an active metal, whereby the catalyzer is maintained in suspension and the acid or ester and hydrogen are brought into intimate contact with the active surfaces of said catalyzer.

2. The process of hydrogenizing fatty acids and their esters, which consists in mechanically subdividing and agitating, in the presence of an atmosphere of hydrogen, a heated mixture of the fatty acid or ester and a pulverulent catalyzer consisting of a light, voluminous, finely-divided, inert support impregnated with metallic nickel, whereby the catalyzer is maintained in suspension and the acid or ester and hydrogen are brought into intimate contact with the active surfaces of said catalyzer.

3. The process of hydrogenizing fatty acids and their esters, which consists in repeatedly projecting or propelling into an atmosphere of compressed hydrogen portions of a heated mixture of the fatty acid or ester and a pulverulent catalyzer consisting of a light, voluminous, finely-divided, inert support impregnated with an active metal.

EDWIN JUNO KAYSER.

Attest:
ARTHUR H. EWALD,
EARL W. GRIFFIN.

Correction in Letters Patent No. 1,008,474.

It is hereby certified that the name of the assignee in Letters Patent No. 1,008,474, granted November 14, 1911, upon the application of Edwin Cuno Kayser, of Cincinnati, Ohio, for an improvement in "Methods of Saturating Fatty Acids or Their Glycerids With Hydrogen," was erroneously written and printed "The Proctor and Gamble Company," whereas it should have been written and printed *The Procter and Gamble Company;* and that the said Letters Patent should be read with this correction therein that the same may conform to the record of the case in the Patent Office.

Signed and sealed this 16th day of January, A. D., 1912.

[SEAL.]

C. C. BILLINGS,

Acting Commissioner of Patents.