

PATENT SPECIFICATION

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(54) IMPROVED PROCESS FOR EXTRACTING VANADIUM FROM DEACTIVATED CATALYSTS

(71) We, SHELL INTERNATIONALE RESEARCH MAATSCHAPPIJ B.V., a company organised under the laws of The Netherlands, of 30 Carel van Bylandtlaan, The Hague, The Netherlands, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

Patent 1,526,927 to which the present application is an addition, relates to a process for extracting vanadium from a catalyst which has been deactivated by use in the treatment of a hydrocarbon oil containing vanadium with hydrogen at elevated temperature and pressure, during which treatment the vanadium content of the catalyst has increased by at least 10 pbw. According to the said patent 1,526,927 the extraction of the vanadium, whereby the vanadium content of the catalyst is decreased by at least 40% of the amount by which it has risen during the deactivation, is carried out by extracting the deactivated catalyst with an aqueous solution of a mineral acid, after which vanadium is separated from the vanadium-containing solution thus obtained. If the process is applied to a catalyst that has been deactivated in the hydrotreatment of a hydrocarbon oil containing nickel in addition to vanadium, during which treatment the nickel content of the catalyst has increased as well, nickel is also removed from the catalyst in the process. Besides the extraction of vanadium and optionally nickel from deactivated catalysts, the process is also applicable to the regeneration of deactivated catalysts so that they can be used again for catalytic purposes.

According to the said Patent 1,526,927 the acid extraction is preferably carried out in the presence of a reducing agent.

Also, according to the said patent 1,526,927 it is preferred to treat the deactivated catalyst first with steam to remove sulphur and then with an oxygen-containing gas to remove carbon, prior to subjecting it to acid extraction. It should be remarked that, when the aim is not only to extract vanadium and optionally nickel from the catalyst, but also to regenerate the catalyst, the treatment of the deactivated catalyst with an oxygen-containing gas in the way indicated in the examples of the said patent 1,526,927 whereby a small portion of the deactivated catalyst is treated with air for three hours at 550°C, is not suitable for larger quantities of deactivated catalyst because of the large amount of heat that would be liberated.

When larger quantities of deactivated catalyst had to be treated with a view to extracting vanadium and optionally nickel from the catalyst as well as regenerating the catalyst, the following three-stage procedure was until recently considered to be the most attractive embodiment of the process according to the said patent 1,526,927. The deactivated catalyst is first treated for 1-5 hours at 250-450°C and atmospheric pressure with a mixture of steam and nitrogen, then for 1 - 5 days at 350 - 600°C and atmospheric pressure with a mixture of air and nitrogen and finally it is subjected to acid extraction in the presence of a reducing agent for 0.5-3 hours at 50-150°C. The treatment times required in the various stages are dependent upon, inter alia, the quantities of sulphur, carbon and metals which are present on the deactivated catalyst and the conditions chosen, viz. treatment temperatures, gas flow rates and compositions of treating gases and extraction liquid. Up to now the long treatment time required in the second stage of the three-stage pro-

cedure has been considered a serious drawback for using the process according to the said patent 1,526,927 on a commercial scale.

- 5 Continued investigation of the process as described in the said patent 1,526,927 has now led to the finding that a comparable result can be obtained as regards vanadium removal and activity of the re-
- 10 generated catalyst to that achieved by conducting the process according to the three-stage procedure described hereinbefore, but in a much shorter time, if the acid extrac-
- 15 tion is preceded by treatment of the deactivated catalyst with steam at a temperature above 250°C and a pressure above 1.5 bar. In addition to the fact that with the process now found a much shorter treatment time will suffice for achieving
- 20 a comparable result as regards vanadium removal and activity of the regenerated catalyst, this process has three additional advantages over the above-mentioned three-stage procedure in that the number
- 25 of treatment stages preceding the acid extraction has decreased by one, the number of gases required for treating the deactivated catalyst prior to acid extraction has been reduced to one and a reducing agent
- 30 is no longer used in the acid extraction.

The present patent application therefore relates to an improved process for extracting vanadium from a deactivated catalyst according to the patent 1,526,927,

- 35 in which process vanadium is extracted from a catalyst which has been deactivated by use in the treatment of a vanadium-containing hydrocarbon oil with hydrogen at elevated temperature and pressure,
- 40 during which treatment the vanadium content of the catalyst has increased by at least 10 pbw, and in which process the said vanadium extraction, whereby the vanadium content of the catalyst is re-
- 45 duced by at least 40% of the amount by which it has risen during the deactivation (vanadium content of the catalyst expressed in pbw vanadium/100 pbw catalyst carrier), is carried out by extracting the deactivated
- 50 catalyst with an aqueous solution of a mineral acid (called hereinafter "acid extraction"), after which the vanadium is separated from the vanadium-containing solution thus obtained.

- 55 The improvement consists in that the acid extraction is preceded by a treatment of the deactivated catalyst with steam at a temperature above 250°C and a pressure above 1.5 bar.

- 60 In the process according to the present invention the treatment with steam is conducted at a temperature above 250°C, but preferably below 550°C and in particular between 325 and 450°C. The steam treat-
- 65 ment is performed at a pressure above 1.5

bar, but preferably below 10 bar and in particular between 2 and 7 bar.

The acid extraction to which the deactivated catalyst should be subjected in the process according to the invention, is preferably carried out at elevated temperature, in particular at a temperature above 50°C. The acid extraction is preferably conducted with an aqueous solution of sulphuric acid.

In the catalytic hydrotreatment of hydrocarbon oils at elevated temperature and pressure, carbon is deposited on the catalyst during the initial period until a certain carbon content has been reached, after which the carbon content of the catalyst remains practically constant during the further course of the operation. When the results of the process now proposed are compared with those of the three-stage procedure discussed hereinbefore, it is found that carbon removal as in the three-stage procedure does not take place now. It also appears that catalysts regenerated according to the present invention, in spite of the carbon present on them, have an activity comparable to that of carbon-free catalysts which have been regenerated according to the three-stage procedure. If desired, one may remove carbon from the catalysts also in the process according to the invention by treating the catalysts after the acid extraction at elevated temperature with an oxygen-containing gas. In comparison with the second stage of the three-stage procedure, in which carbon is also removed from the catalyst, the treatment with an oxygen-containing gas, optionally to be included in the process according to the invention, is a much simpler step, because the amount of heat released in this treatment is much smaller.

In the catalytic hydrotreatment at elevated temperature and pressure of the hydrocarbon oils containing nickel besides vanadium, nickel is also deposited on the catalyst. Comparison of the results of the process now proposed with those of the three-stage procedure discussed hereinbefore shows that nickel is removed to a lesser extent now than in the three-stage procedure. It is also found that catalysts regenerated according to the present invention, in spite of the nickel present on them, have an activity comparable to that of practically nickel-free catalysts which have been regenerated according to the three-stage procedure. If desired, one may remove more nickel from the catalysts in the process according to the invention. This can very suitably be done by treating the regenerated catalyst first at elevated temperature with an oxygen-containing gas and then subjecting it again to an acid extraction.

Hydrocarbon oils which contain nickel besides vanadium and which in the hydro-treatment at elevated temperature and pressure cause both vanadium and nickel deposition on the catalyst, contain as a rule much more vanadium than nickel. The quantity of nickel deposited on the catalyst is therefore as a rule only a fraction of the quantity of vanadium deposited. When a catalyst is alternately used for hydrotreating a vanadium-and nickel-containing hydrocarbon oil at elevated temperature and pressure and is regenerated according to the invention, the nickel content may in the long run build up to an undesirably high value. This can be prevented in the regeneration by subjecting a slipstream of the regenerated catalyst successively to a treatment with an oxygen-containing gas at elevated temperature and an acid extraction. The catalyst slipstream from which the nickel has been removed is then added to the main stream of the regenerated nickel-containing catalyst. The process according to the invention in which nickel build-up on the catalyst is prevented by subjecting a slipstream of the regenerated catalyst to an additional treatment may also be carried out as follows. The regenerated catalyst is subjected at elevated temperature to a treatment with an oxygen-containing gas for the removal of carbon. From the catalyst thus treated a slipstream is separated, which is subjected to an acid extraction. The catalyst slipstream from which nickel has been removed is then added to the main stream of the nickel-containing catalyst from which carbon has been removed.

The process according to the present invention is particularly important in those cases in which the aim is not only to extract vanadium and optionally nickel from the deactivated catalyst, but also to regenerate the catalyst (which may, in the fresh condition, contain one or more metals with hydrogenation activity) so that it can be used again for catalytic purposes. The present patent application relates therefore not only to a process for extracting vanadium and optionally nickel from a deactivated catalyst, but also to a process in which this extraction is conducted in such a way that a regenerated catalyst is obtained which can be used again for catalytic purposes, either as such, or after a complementary quantity of metals with hydrogenation activity has been added to it. The process according to the invention is especially important for extracting vanadium and optionally nickel from a catalyst substantially consisting of silica, in combination with regeneration of the catalyst, which catalyst has been used in a process for the hydrodemetallization of a

hydrocarbon oil.

Applicants copending application (Serial No. 1 567 140) 11405/78 also relates to an improved process for extracting vanadium from deactivated catalysts.

The invention will now be explained with reference to the following examples.

EXAMPLE I

A catalyst comprising 0.5 pbw nickel and 2.0 pbw vanadium per 100 pbw silica carrier was prepared by impregnating a silica carrier with an aqueous solution of nickel nitrate and vanadyl oxalate, after which the composition was dried and calcined. The catalyst (catalyst A) was used in the sulphidic form for the hydrodemetallization of a hydrocarbon oil (oil A) with a total vanadium and nickel content of 62 ppmw, a C-asphaltenes content of 6.4 %w and a sulphur content of 3.9 %w, which oil had been obtained as the residue in the atmospheric distillation of a crude oil from the Middle East. The hydrodemetallization was carried out by passing the oil together with hydrogen in a downward direction through a cylindrical, vertically disposed fixed catalyst bed at a temperature of 420°C, a total pressure of 150 bar, a space velocity of 5 kg.l⁻¹.h⁻¹ and a gas flow rate (measured at the reactor outlet) of 250 NI H₂.kg⁻¹. The activity of the catalyst, expressed as "% vanadium removed" (= average vanadium removal over the period of catalyst age from 1 tonne oil/kg catalyst to 4 tonnes oil/kg catalyst), was 51. After the catalyst had been deactivated in this process, it was extracted with toluene to remove remnants of residual oil and after evaporation of the toluene from the catalyst the latter was analysed. The deactivated catalyst (catalyst B) contained 9.7 pbw carbon, 20.6 pbw sulphur, 4.1 pbw nickel and 24.3 pbw vanadium per 100 pbw silica.

EXAMPLE II

5 kg of Catalyst B was treated with a 4:1 steam/nitrogen mixture for three hours at 350°C, atmospheric pressure and a gas flow rate of 2NI gas mixture.(g catalyst)⁻¹.h⁻¹. The catalyst was then treated with a 1:9 air/nitrogen mixture for 50 hours at 400°C, atmospheric pressure and a gas flow rate of 1 NI gas mixture.(g catalyst)⁻¹.h⁻¹. Finally, the catalyst was extracted for two hours at 90°C with stirring with 40 l 2 N sulphuric acid which had been saturated with sulphur dioxide. After the extracted catalyst had been washed with water, it was dried at 120°C and calcined for three hours at 550°C. On analysis of the catalyst thus obtained (catalyst C), 96% of the vanadium and 95% of the nickel were found to have been removed from the catalyst by this treatment.

EXAMPLE III

5 kg of Catalyst B was treated with steam for five hours at a temperature of 350°C, a pressure of three bar and a space velocity of 2.6 kg steam.(kg catalyst⁻¹.h⁻¹). The catalyst was then extracted for two hours at 90°C with stirring with 40 l 2 N sulphuric acid. After the extracted catalyst had been washed with water it was dried at 120°C. On analysis of the catalyst thus obtained (catalyst D), 95% of the vanadium and 45% of the nickel were found to have been removed from the catalyst by this treatment.

EXAMPLE IV

5 kg of Catalyst B was treated in much the same way as in Example III, but now the steam treatment was conducted at a temperature of 425°C. On analysis of the catalyst thus obtained (catalyst E), 90% of the vanadium and 60% of the nickel were found to have been removed from the catalyst by this treatment.

EXAMPLE V

5 kg of Catalyst B was treated in much the same way as in Example III, but now the steam treatment was conducted at a pressure of 6 bar. On analysis of the catalyst thus obtained (catalyst F), 93% of the vanadium and 57% of the nickel were found to have been removed from the catalyst by this treatment.

EXAMPLE VI

5 kg of Catalyst B was treated in much the same way as in Example III, but now the acid extraction was conducted for one hour with 20 l 4 N sulphuric acid. On analysis of the catalyst thus obtained (catalyst G), 96% of the vanadium and 60% of the nickel were found to have been removed from the catalyst by this treatment.

EXAMPLE VII

5 kg of Catalyst B was treated in much the same way as in Example III, but now the acid extraction was conducted for one hour with 20 l 6 N sulphuric acid. On analysis of the catalyst thus obtained (catalyst H), 96% of the vanadium and 75% of the nickel were found to have been removed from the catalyst by this treatment.

EXAMPLE VIII

5 kg of Catalyst D which, like catalysts B, E, F, G and H, contained 9.7 pbw carbon per 100 pbw silica, was treated with a 1:5 air/nitrogen mixture for 10 hours at 400°C, atmospheric pressure and a gas flow rate of 1 NI gas mixture.(g catalyst)⁻¹.h⁻¹. Analysis of the catalyst thus obtained (catalyst I) showed that it contained only 0.1 pbw carbon per 100 pbw silica.

EXAMPLE IX

5 kg of Catalyst I was extracted for two hours at 90°C with 5 l 2 N sulphuric acid. After the extracted catalyst had been

washed with water it was dried at 120°C and calcined for three hours at 550°C. Analysis of the catalyst thus obtained (catalyst J) showed that relative to the deactivated catalyst B 99% of the vanadium and 99% of the nickel had been removed from the catalyst.

EXAMPLE X

5 kg of Catalyst B was treated in much the same way as in Example III, but now the steam treatment was conducted at a temperature of 200°C and a pressure of 4 bar. On analysis of the catalyst thus obtained (catalyst K), 46% of the vanadium and 35% of the nickel were found to have been removed from the catalyst by this treatment.

EXAMPLE XI

5 kg of Catalyst B was treated in much the same way as in Example III, but now the steam treatment was conducted at a pressure of 0.5 bar. On analysis of the catalyst thus obtained (catalyst L), 71% of the vanadium and 43% of the nickel were found to have been removed from the catalyst by this treatment.

EXAMPLE XII

5 kg of Catalyst B was treated in much the same way as in Example III, but now the steam treatment was conducted at a pressure of 1.0 bar. On analysis of the catalyst thus obtained (catalyst M), 80% of the vanadium and 43% of the nickel were found to have been removed from the catalyst by this treatment.

EXAMPLE XIII

A catalyst containing 0.5 pbw nickel and 2.0 pbw vanadium per 100 pbw silica carrier was prepared by impregnating catalyst C with an aqueous solution of nickel nitrate and vanadyl oxalate, after which the composition was dried. The catalyst C thus obtained was used in the sulphidic form of the hydrodemetallization of oil A under the same conditions as the hydrodemetallization of this oil with catalyst A described in Example I. The activity of catalyst C, expressed as "% vanadium removed", was 48.

EXAMPLE XIV

Catalysts containing 1.0 to 2.8 pbw nickel and 2.0 pbw vanadium per 100 pbw silica carrier were prepared by impregnating catalysts D, E, F, G, H and I with an aqueous solution of vanadyl oxalate, after which the compositions were dried. Catalysts D', E', F', G', H' and I' thus obtained were used for the hydrodemetallization of oil A in the same way as described in Example XIII. The activities of catalysts D', E', F', G', H' and I', expressed as "% vanadium removed", were 47, 48, 49, 48, 49 and 51, respectively.

EXAMPLE XV

In the same way as described in Example

XIII catalyst J' was prepared from catalyst J and used for the hydrodemetallization of oil A. The activity of catalyst J', expressed as "percentage vanadium removed", was 51.

Of the Examples I-XV Nos. III-IX, XIV and XV are examples according to the present invention. The other examples have been included for comparison.

Example I relates to a hydrodemetallization in which a fresh catalyst A deactivates to the deactivated catalyst B.

Example II relates to the three-stage procedure described hereinbefore in which a regenerated catalyst C is prepared from the deactivated catalyst B and in which it takes 53 hours in all to carry out the first two stages.

Examples III-VII relate to the improved process according to the invention in which regenerated catalysts D-H are prepared from the deactivated catalyst B. Mutual comparison of Examples II-VII shows that the process according to the invention leads to the same excellent vanadium removal as the three-stage procedure. However, the steam treatment at a pressure above 1.5 bar, which has replaced the first and the second stage of the three-stage procedure, takes only five hours.

Example VIII relates to the treatment with an oxygen-containing gas at elevated temperature of the carbon- and nickel-containing regenerated catalyst H, yielding catalyst I from which the carbon has been removed. Example IX relates to the acid extraction of the nickel-containing regenerated catalyst I, yielding catalyst J from which the nickel has been removed.

Examples X-XII relate to the treatment of the deactivated catalyst with steam followed by acid extraction, in which "regenerated catalysts" K-M are prepared from deactivated catalyst B. During the treatment with steam the temperature was too low in Example X and the pressure too low in Examples XI and XII. This resulted in an insufficient vanadium removal for catalysts K-M (and consequently in a low activity!).

From Examples XIII-XV it is seen that catalysts which have been regenerated according to the present invention (catalysts D'-J') show the same high activity as a catalyst which has been regenerated according to the three-stage procedure (catalyst C').

WHAT WE CLAIM IS:—

1. An improved process for extracting vanadium from a deactivated catalyst according to patent 1,526,927 in which process vanadium is extracted from a catalyst which has been deactivated by use in the treatment of a vanadium-containing hydrocarbon oil with hydrogen at elevated

temperature and pressure, during which treatment the vanadium content of the catalyst has increased by at least 10 pbw, and in which process the said vanadium extraction, whereby the vanadium content of the catalyst is reduced by at least 40% of the amount by which it has risen during the deactivation (vanadium content of the catalyst expressed in pbw vanadium/100 pbw catalyst carrier), is carried out by extracting the deactivated catalyst with an aqueous solution of a mineral acid (called herein: "acid extraction"), after which the vanadium is separated from the vanadium-containing solution thus obtained, wherein

the improvement consists in that the acid extraction is preceded by a treatment of the deactivated catalyst with steam at a temperature above 250°C and a pressure above 1.5 bar.

2. A process according to claim 1, characterized in that the treatment of the deactivated catalyst with steam is conducted at a temperature below 550°C.

3. A process according to claim 2, characterized in that the said temperature is between 325 and 425°C.

4. A process according to any one of claims 1-3, characterized in that the treatment of the deactivated catalyst with steam is conducted at a pressure below 10 bar.

5. A process according to claim 4, characterized in that the said pressure is between 2 and 7 bar.

6. A process according to any one of claims 1-5, characterized in that the acid extraction is carried out at a temperature above 50°C.

7. A process according to any one of claims 1-6, characterized in that the acid extraction is carried out with an aqueous solution of sulphuric acid.

8. A process according to any one of claims 1-7, characterized in that carbon is removed from the catalyst by treating the latter after the acid extraction with an oxygen-containing gas at elevated temperature.

9. A process according to claim 8, characterized in that nickel is removed from the catalyst by subjecting the latter again to an acid extraction after the treatment with an oxygen-containing gas.

10. A process according to claim 8, characterized in that for the removal of nickel a slipstream of the catalyst treated at elevated temperature with an oxygen-containing gas is again subjected to an acid extraction and that the catalyst slipstream from which the nickel has been removed is added to the main stream of the nickel-containing catalyst from which carbon has been removed.

11. A process according to any one of

XIII catalyst J' was prepared from catalyst J and used for the hydrodemetallization of oil A. The activity of catalyst J'; expressed as "percentage vanadium removed", was 51.

Of the Examples I-XV Nos. III-IX, XIV and XV are examples according to the present invention. The other examples have been included for comparison.

Example I relates to a hydrodemetallization in which a fresh catalyst A deactivates to the deactivated catalyst B.

Example II relates to the three-stage procedure described hereinbefore in which a regenerated catalyst C is prepared from the deactivated catalyst B and in which it takes 53 hours in all to carry out the first two stages.

Examples III-VII relate to the improved process according to the invention in which regenerated catalysts D-H are prepared from the deactivated catalyst B. Mutual comparison of Examples II-VII shows that the process according to the invention leads to the same excellent vanadium removal as the three-stage procedure. However, the steam treatment at a pressure above 1.5 bar, which has replaced the first and the second stage of the three-stage procedure, takes only five hours.

Example VIII relates to the treatment with an oxygen-containing gas at elevated temperature of the carbon- and nickel-containing regenerated catalyst H, yielding catalyst I from which the carbon has been removed. Example IX relates to the acid extraction of the nickel-containing regenerated catalyst I, yielding catalyst J from which the nickel has been removed.

Examples X-XII relate to the treatment of the deactivated catalyst with steam followed by acid extraction, in which "regenerated catalysts" K-M are prepared from deactivated catalyst B. During the treatment with steam the temperature was too low in Example X and the pressure too low in Examples XI and XII. This resulted in an insufficient vanadium removal for catalysts K-M (and consequently in a low activity!).

From Examples XIII-XV it is seen that catalysts which have been regenerated according to the present invention (catalysts D'-J') show the same high activity as a catalyst which has been regenerated according to the three-stage procedure (catalyst C).

WHAT WE CLAIM IS:—

1. An improved process for extracting vanadium from a deactivated catalyst according to patent 1,526,927 in which process vanadium is extracted from a catalyst which has been deactivated by use in the treatment of a vanadium-containing hydrocarbon oil with hydrogen at elevated

temperature and pressure, during which treatment the vanadium content of the catalyst has increased by at least 10 pbw, and in which process the said vanadium extraction, whereby the vanadium content of the catalyst is reduced by at least 40% of the amount by which it has risen during the deactivation (vanadium content of the catalyst expressed in pbw vanadium/100 pbw catalyst carrier), is carried out by extracting the deactivated catalyst with an aqueous solution of a mineral acid (called herein: "acid extraction"), after which the vanadium is separated from the vanadium-containing solution thus obtained, wherein

the improvement consists in that the acid extraction is preceded by a treatment of the deactivated catalyst with steam at a temperature above 250°C and a pressure above 1.5 bar.

2. A process according to claim 1, characterized in that the treatment of the deactivated catalyst with steam is conducted at a temperature below 550°C.

3. A process according to claim 2, characterized in that the said temperature is between 325 and 425°C.

4. A process according to any one of claims 1-3, characterized in that the treatment of the deactivated catalyst with steam is conducted at a pressure below 10 bar.

5. A process according to claim 4, characterized in that the said pressure is between 2 and 7 bar.

6. A process according to any one of claims 1-5, characterized in that the acid extraction is carried out at a temperature above 50°C.

7. A process according to any one of claims 1-6, characterized in that the acid extraction is carried out with an aqueous solution of sulphuric acid.

8. A process according to any one of claims 1-7, characterized in that carbon is removed from the catalyst by treating the latter after the acid extraction with an oxygen-containing gas at elevated temperature.

9. A process according to claim 8, characterized in that nickel is removed from the catalyst by subjecting the latter again to an acid extraction after the treatment with an oxygen-containing gas.

10. A process according to claim 8, characterized in that for the removal of nickel a slipstream of the catalyst treated at elevated temperature with an oxygen-containing gas is again subjected to an acid extraction and that the catalyst slipstream from which the nickel has been removed is added to the main stream of the nickel-containing catalyst from which carbon has been removed.

11. A process according to any one of





claims 1-10, characterized in that it is applied to a catalyst substantially consisting of silica, which catalyst has been used in a process for the hydrodemetallization of a hydrocarbon oil.

12. A process for extracting vanadium and optionally nickel from a deactivated catalyst, substantially as described hereinbefore and in particular with reference to Examples III-IX, XIV and XV.

13. Vanadium and optionally nickel extracted from a deactivated catalyst accord-

ing to a process as described in claim 12. Catalysts that have been regenerated according to a process as described in claim 12.

15. Catalytic processes in which a catalyst according to claim 14 is used.

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