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VANADIUM COMPOUND AND METHOD OF MAKING SAME.

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This invention relates to the art of recovery of metals, and particularly has reference to the recovery of vanadium from a product resulting from the combustion or burning of petroleum.

I have discovered that the residue, or product of combustion, resulting from the burning of petroleum, contains vanadium, the said residue, or product, being commonly known as soot or flue-dust which collects in the stacks or chimneys of buildings, ships, furnaces and in the retorts and on the fire sides of the boilers and tubes of oil burning apparatus and installations. The burning of petroleum also produces a substance, which is neither soot nor flue-dust, as a deposit in the retorts, on the boilers, tubes, etc., of furnaces, which also contains vanadium in a high percentage; but, I desire to have it understood that all forms of deposits, resulting from the burning of petroleum, whether in the form of soot, flue-dust, scale, or other substance, are within the contemplation of my invention and are to be understood to be included in my claims. That is to say, in order to simplify my description and claims, I will use the term "soot" to comprehend and include all the forms of deposit resulting from the combustion of petroleum, regardless of the apparatus in which the oil is burned. I have discovered, also, that the said deposit contains radium and uranium; but, since, according to my discovery, vanadium is found in higher percentage, my invention primarily relates to the recovery of vanadium, and the process with which I have experimented is directed more particularly to the recovery of vanadium than of the other metals.

In order to demonstrate my discovery, I have subjected large quantities of petroleum soot to treatment and have been successful in obtaining vanadium compounds therefrom, together with alloys thereof. Therefore, my invention consists in the method or process of recovering vanadium from petroleum soot, and in the product of said process, both immediate and mediate, as hereinafter described and claimed.

The process which I have employed in the recovery of vanadium is as follows:

I first transform the bulk soot into briquettes. This reduces the bulk and enables the substance to be conveniently and properly handled. Or, first, I may roast the soot in a rotary kiln; but, I conceive that the roasting may be carried out by other means or apparatus. This roasting eliminates most of the sulphur and carbon and markedly reduces the material both in bulk and weight.

To transform the bulk, either roasted or unroasted, into briquettes, I mix the bulk with a binder in suitable proportions, such as sodium silicate solution, and subject the mixture to pressure, or its equivalent, in a suitable mold or chamber, with or without the presence of heat. In this connection, I place no limit upon the size of the briquette, or cake, or block, formed, since the limitations imposed, in any event, will have reference to convenience and facility in handling. When the briquette is formed or made if the bulk has not been roasted, the briquette may be roasted. I wish it understood, however, that it is not an essential of my process that the bulk or briquette should be roasted; nor is it an essential of my process that the bulk material should be transformed into briquettes, blocks, or cakes.

The roasted or unroasted material or briquette is then fused to bring the vanadium into solution; or it may be leached at desired temperature with either acid or alkaline solution. As an example of a means for this purpose I may use a fusion with approximately three or four times its weight of niter cake. The fused mass, after cooling, is broken up and leached with water. The solution thus produced is filtered from the insoluble residue. The filtered solution will contain a large percentage of the vanadium present in the soot.

The solution is treated with hydrogen sulphide to remove molybdenum, copper and other metals of the hydrogen sulphide group which may be present. The precipitated sulphides are removed by filtration and the clear solution is heated, air being blown in to remove the H_2S.

The solution thus obtained is then treated generally according to the process prescribed by the Bureau of Mines in its Bulletin 104, Mineral Technology 12, for the extraction and recovery of vanadium from carnotite, said further steps being substantially as follows:

The solution, containing the vanadium and any uranium present, is run slowly with

constant stirring into a boiling solution of sodium carbonate. The carbonate solution should be of such strength that the final solution is strongly alkaline. The amount of excess carbonate will vary with the amounts of vanadium and uranium and may range from 150 to 600 lbs. per ton of roasted soot treated. The solution is boiled for from two to three hours after the final addition of the blue acid solution to completely precipitate the iron, cromium, aluminum, etc., and is then filtered. If the amount of excess carbonate and the time of boiling are carefully checked and controlled the precipitate will be reasonably free from vanadium. The filtrate from the iron precipitate is neutralized with HNO_3, the solution being left just neutral. Air is blown through the boiling solution to remove CO_2. A solution of ferrous sulphate is added to precipitate the vanadium as iron vanadate. Since the addition of the sulphate will leave the solution slightly acid, enough NaOH is added to again make it neutral. The precipitate will be yellow, brown or gray-green according to the conditions under which the precipitation was made and will contain from 25 to 40 per cent of V_2O_5. The solution is filtered and the iron vanadate precipitate washed and dried. The precipitate may be reduced to ferrovanadium or may be treated by other suitable means to secure other vanadium compounds or alloys, which are in a form for commercial use.

By variations or modifications of the foregoing process, I may also obtain oxide of vanadium or vanadate precipitate, and other compounds and alloys of vanadium, as an article of manufacture or commercial product, my particular product resulting from the foregoing process being, as just stated, ferrovanadium.

I may also produce a concentrate containing vanadium by suspending the soot in water, or producing a water and soot mixture, to render it limpid and capable of being readily poured or of flowing. The limpid mixture is then put through a classifier, the overflow carrying off the soot and lighter materials; that is to say, the specific gravity of the different constituents is utilized to separate the same. I then run the precipitate over a concentrator. The product is a concentrate containing vanadium.

In the foregoing I have set forth the process employed by me in the recovery of vanadium from petroleum soot, and desire to lay stress thereon and upon the products thereof in addition to my primary discovery of vanadium in the soot of petroleum resulting from combustion thereof. I also desire to lay stress upon my primary product, viz, briquette, or block, vanadium-containing soot which gives immediate commerciality to my discovery, since it enables me to convey the vanadium-containing soot directly to the users of large quantities of soot for the production of commercial vanadium, and in this connection I desire it understood that I realize that more satisfactory commercial results may be obtained in a commercialized plant, such as a direct smelter, than I have obtained as the result of the limitations imposed upon me in carrying out and developing my invention resulting from my primary and very important discovery.

Having thus described my invention, what I claim and desire to secure by Letters Patent is:

1. As an article of manufacture, a residue of the products of combustion of petroleum characterized by its content of vanadium.

2. As an article of manufacture, a residue of the products of combustion of petroleum characterized by its content of ferrovanadium.

3. As an article of manufacture, a residue of the products of combustion of petroleum characterized by its content of oxide of vanadium.

4. The method of deriving vanadium precipitate from the products of combustion of petroleum, including treating said products by concentration.

5. As a new article of manufacture a briquette composed of the soot resulting from the burning or combustion of petroleum.

6. The method of recovering vanadium-bearing substances from the products of combustion of petroleum by means of mechanical concentration.

7. The method of deriving vanadium precipitate from petroleum, comprising the production of soot from the petroleum and chemically treating the soot to produce a precipitate.

8. The method of recovering vanadium or a precipitate thereof from petroleum, comprising producing soot from petroleum by burning the latter; and fusing and leaching the soot to produce ferrovanadium, oxide of vanadium, a vanadate precipitate, and other compounds.

9. The method herein described of recovering vanadium from petroleum soot, comprising roasting the soot; fusing the roasted material with niter cake; leaching the fused mass; and filtering the solution.

10. The method herein described of producing a vanadium compound from petroleum soot, comprising producing a limpid mixture of soot and water; subjecting the limpid mixture to the action of a classifier; and subjecting the precipitate to the action of a concentrator, substantially as described.

11. As a new article of manufacture a briquette composed of petroleum soot and a binder.

12. The method of making a briquette of

soot of petroleum comprising mixing the soot with a binder, and subjecting the mixture to pressure to give it a definite form.

13. The method of making a briquette of soot of petroleum comprising mixing the soot with a binder, and molding the mixture into predetermined form.

14. A process for recovering vanadium from petroleum hydrocarbons, consisting in exhausting hydrocarbon products by combustion and collecting the deposited vanadium compounds.

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