United States Patent Office.

CAMPBELL MORFIT, OF NEW YORK, N. Y.

Letters Patent No. 106,851, *dated August* 30, 1870.

IMPROVEMENT IN THE TREATMENT OF GUANO AND THE MANUFACTURE OF FERTILIZERS.

The Schedule referred to in these Letters Patent and making part of the same.

To all whom it may concern.

Be it known that I, CAMPBELL MORFIT, consulting chemist, of the city of New York, in the State of New York and United States of America, but now residing temporarily at Sudbrook Park, Petersham, county of Surrey, England, have invented a new, improved, and economical Method of Treating Navaza Guano and other kinds of Mineral Phosphates, for their Conversion into Valuable Fertilizing Products; and I do hereby declare that the following is a full and exact description thereof.

The nature of my invention consists in the use of crude ammonia, in the form of coal-gas liquor, or as gas distilled therefrom, for recovering back the hydrochloric acid which may be employed for making solutions of navaza (navassa) guano and other kinds of mineral phosphates, by neutralizing the acid into chloride of ammonium, a valuable salt, and simultaneously precipitating the contained phosphate of lime in the state of a very potential fertilizer.

To carry out the invention practically, the navaza guano or mineral phosphate must be reduced first to coarse or fine powder, according to its greater or lesser solubility in acids. It is then placed in stone vats, each of which is fitted with a false bottom, and drenched with commercial hydrochloric acid, after which the vat is to be covered. From this cover there must be a tube connection with a low chamber containing lime, for the condensation of the noxious gases which may be evolved during the action of the acid on the mineral. As this acid percolates downward through the powder, it takes up all the soluble matter, and passes through the false bottom into the chamber below as a strong solution. Time would be saved by keeping the room at 80° to 100° Fahrenheit by means of steam-pipes around the outside of the vats. The acid solution, as it collects in the lower chambers, must be drawn off through a hole in the front, which is fitted with a plug for the purpose. A gutter running the length of the front of these vats must be arranged to conduct the solution into a stone reservoir constructed in the ground beneath, and as a Montejus. The first dose of acid having run through, a fresh addition is next to be poured on, and allowed to pass through in the same way. Finally, and in like manner, two successive additions of boiling water are then to follow, in order to rinse the powder, and the wash waters are made to run and mix with the previous liquors. The residue in the vats is thus exhausted, and must be shoveled out as insoluble matter without value in this process. The vats are then ready to be charged again, with fresh materials for a new operation. The collected liquors in the Montejus are to be raised into a very capacious reservoir of oak wood, by means of compressed air or steam. The pipe through which the liquor is forced from the Montejus up into the reservoir must be of iron, lined with gutta-percha or vulcanized India rubber.

The quantity of acid required for exhausting the mineral must be determined in advance, by testing a weighed portion of the powder, for it will vary with the composition of the phosphatic guano or mineral under treatment. The liquor which has been raised into the vat contains all the matters of the mineral soluble in the hydrochloric acid and water, and consisting of phosphates of lime and magnesia, chlorides of the calcium and magnesium which existed originally as carbonates, together with more or less of alumina and oxide of iron, and some little of phosphates of alumina and iron, if these latter were present. The insoluble residue in the vat is composed of sand, earthy and metallic silicates, fluoride of calcium, much of the alumina and oxide of iron, and nearly the whole of the phosphates of alumina and iron. This insoluble residue is to be drawn out from the vat through a man-hole, and thrown aside as rejected matter. To return to the liquor in the vat, the latter must be fitted with a tight cover, from which a tube leads down the outside of the vat, and along the ground, into the fire of the steam-boiler furnace, in order that the noxious gases which are about to be eliminated may be consumed, for the protection of the comfort and health of the workmen. The vat should be fitted with a wooden stirrer, put in motion by steam gearing, and it should not be filled with liquor to more than one-half of its height, as upper space must be left for the addition of the precipitant and the effervescence which will accompany the chemical action about to take place. Crude gas liquor is now added slowly and by instalments, while the stirrer is in motion, through a funnel in the top of the vat, until the liquor becomes slightly alkaline to test-paper. This test indicates that all the hydrochloric acid of the liquor has been neutralized by the ammonia of the gas liquor, which change causes it to drop at the same time the phosphate of lime, together with any alumina and oxide of iron and their phosphates which the liquor may have contained. When this has settled to the bottom, the liquor above will be an impure solution of chloride of ammonium, and is to be drawn off and evaporated to a dense solution, in order that it may form into crystals. These crystals of crude chloride of ammonium may be purified by recrystallization, or else baked on a carefully-heated drying-kiln, so as to destroy its empyzeumatic, sulphureted, and foreign organic matters, and then sent into market as ammoniacal salt for the manufacture of fertilizers. The remaining phosphate of lime retains a considerable portion of the impure chloride of ammonium liquor, and is colored more or less light greenish blue by the

small quantities of metallic sulphurets which have been formed by the impurities of the two liquors. There is present, also, some pitchy or empyzeumatic matters. The phosphate of lime is to be drawn from the vat, through wooden gutters, into a kiln similar to that which is used for drying white lead. By a carefully-managed heat it soon becomes a dry powder, of a bluish-grey color, and consisting of gelatinous or precipitated phosphate of lime, with chloride of ammonium. The gelatinous phosphate of lime retains, even as dry powder, a certain amount of water, which may be considered quasi constitutional, and is in that state most suited to give a prompt and consistent fertilizations to soils. Indeed, it has just that condition to which soluble superphosphate of lime is immediately reduced by the natural chemistry of the soil, as soon as it has been applied, and previous to being taken up by the growing crop. To perfect its efficiency as a fertilizer, the ratio of ammoniacal salt should be raised to twenty or thirty per cent., and associated with about ten per cent. of potassa salt, according to the kind of crop for which it may be intended. This is done practically by mixing the proper weights of those salts with the phosphate prepared in manner as above explained.

The amount of crude gas liquor which may be required to neutralize the hydrochloric-acid liquor of the vat may be determined in advance by gauging the quantity of the latter, then taking out a measured portion of it, and carefully adding from a measured portion of the gas liquor until neutralized. From the volume of the latter consumed for this preliminary test sample may be calculated the total required by the contents of the vat. This test is necessary at each operation, because the ammoniacal strength of the crude gas liquor is very variable.

The inconvenience of sulphureted odors from large bulk of liquid, and of color in the precipitated phosphate of lime, may be obviated by mixing the crude gas liquor with lime, and, it may be, also, some oxide of iron, in a round wrought-iron boiler, with a dome top and a tube leading from it into the contents of the vat. Another tube, leading from the steam-boiler, must pass down to the iron still containing the mixture of gas liquor and lime, or lime and oxide of iron.

The cock being opened and steam let on, causes the gas liquor to boil and give off its ammonia as gas, which distils over, with more or less condensed steam, into the vat liquor, to form there, as already explained, chloride of ammonium and precipitated phosphate of lime, but in this instance very much freer from color and disagreeable impurities.

The requisite quantity of gas liquor will be the same, as may be determined by the method above explained. Neutralization of the vat liquor is complete when a piece of red litmus-paper dipped into it becomes immediately and permanently blue.

Claim.

Hydrochloric acid being in the public domain of chemical knowledge as a solvent of phosphatic substances, I do not claim its use for that purpose; but

What I do claim is—

The method of recovering back the hydrochloric acid employed in making hydrochloric solutions of navaza (navassa) guano and other kinds of mineral phosphate of lime, by the use of crude ammonia either in the form of coal-gas liquor or as gas distilled therefrom, for neutralizing the acid, so as to produce simultaneously the salt chloride of ammonium and a semi-gelatinous precipitate of great agricultural value, to be known as Colombian phosphate of lime or fertilizer, by the suitable means and after the economical manner herein described and substantially set forth.

In witness whereof, I, the said CAMPBELL MORFIT, have hereunto set my hand and seal, this 18th day of February, in the year of our Lord 1870.

CAMPBELL MORFIT.

Witnesses:
T. W. FREJON,
F. W. ATKINSON.