## UNITED STATES PATENT OFFICE

2,123,248

INSOLUBLE REACTION PRODUCTS OF PEAT AND PROCESS OF MAKING SAME

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No Drawing. Application January 14, 1937, Serial No. 120,506

8 Claims. (Cl. 167—53)

(Granted under the act of March 3, 1883, as amended April 30, 1928; 370 O. G. 757)

This application is made under the act of March 3, 1883, as amended by the act of April 30, 1928, and the invention herein described and claimed may be manufactured and used by or for the Government for governmental purposes without payment to me of any royalty thereon.

My invention relates to the reaction products of certain organic bases with peat and their method of preparation. The organic bases embraced within the scope of my invention are the stronger titratable bases, and of these particularly those that are appreciably soluble in water. Such bases include aliphatic and aromatic amines, mixed amines, and alkaloids.

One of the objects of my invention is to provide means for rendering these bases less soluble in water, the free bases themselves being soluble. Their usefulness and application are thus increased, as in the field of veterinary medicine where they offer possibilities in the control of internal parasites, when administered in a substantially insoluble form.

When peat and an organic base are brought together in the presence of water a reaction occurs which results in the formation of salts of the base with the acid constituents of the peat. This is a typical acid-base reaction. For this purpose the most acid peats are desirable. However, any of the so-called alkaline or neutral peats which are already neutralized wholly or in part by inorganic bases present in their natural environment, can be brought to the maximum or ideal acid state by treatment with acids. Any of the group of acids consisting of the mineral acids. such as hydrochloric, sulphuric and nitric acids, and acetic acid, is suitable for this purpose. Such treatment dissolves out the inorganic bases, which are chiefly calcium, magnesium, aluminum, iron, 16 etc., and considerably increases the combining power of the peat toward organic bases.

The acidity of peat in its acid or de-based form, as measured in units of pH, is in the neighborhood of 3. It is, therefore, sufficiently acid to combine with organic bases which themselves are definitely alkaline. These bases are titratable with indicators and in general have an alkalinity sufficient to impart a pink color to phenolphthalein, or in other words, a pH greater than 8. Among the bases meeting this requirement are the alkaloids, arecoline, anabasine, and codeine, as well as the simpler amines, such as ethylene diamine and piperidine.

The list of organic bases coming within the 55 scope of my invention is not restricted to those

enumerated, but may be expanded to include any appreciably soluble, strong organic base.

Weak bases, such as pyridine and caffeine, are not capable of combining with peat except to a very small degree and it is not considered practicable to attempt to lower their solubility by this means.

Of the several types of peat that have been tried a New Jersey reed peat, after acid treatment, has been found to be best, in that it fixes the greatest amount of base in insoluble form, other factors being the same. This is the type of peat with which the hereinafter mentioned experiments were conducted. Other satisfactory types of peat, however, include Michigan sedge, Minnesota reed, and German moss peat.

Every reaction between peat and organic base results in the formation of two products: (1) an insoluble product containing most of the base, and (2) a soluble product containing the remainder of the base. The formation of insoluble and soluble compounds of the base with peat appears to be due to the multi-acid nature of peat constituents, of which little is known. In order to convert the maximum amount of base into the insoluble form, an excess of peat is always used. The mixture under these conditions is definitely acid, so that all of the base passes into the salt form. The insoluble product, which contains the greater part of the base used, is separated 30 from the liquid portion, thereby resulting in a product having all of its base substantially insoluble in water.

A typical procedure in preparing these products is as follows: New Jersey peat is de-based by means of hydrochloric acid and thoroughly washed. An aqueous solution of the organic base is added to the peat, with enough water present to form a thin slurry; the mixture is heated on the steam bath for a short time, say 15 minutes, it is then allowed to settle, the liquid poured off, and the solid portion is subsequently washed until the wash water gives only a small test for the base.

The solid product is dried and ground to the desired degree of fineness. The wash waters may be treated for recovery of the base, or they may be evaporated to dryness, thus obtaining the water-soluble product in solid form, known as the "humate". The solid product is known as the "peat" product, thus, "arecoline peat". The peat product is examined for amount of total base and the proportion of total base which is insoluble in water. The humate is examined for total base, all of this being water-soluble. Data obtained 55

from the reaction of peat with several organic bases are given in the following table.

it has unique properties which are not the individual properties of the organic base and peat,

Data pertaining to reaction products of organic bases and

	<i>pour</i>								
10	Base	Peat used	Base used	Peat used to 1 equiva- lent of base, X 0.01	Yield of "peat"	Base in "peat"	Insolubility of base in "peat"	Yield of hu- mate	Base in hu- mate
15	Arecoline Codeine Ethylene dia- mine Piperidine	Grams 20 116 100 100	Grams 3. 6 32 3. 7 10. 5	Grams 8. 6 10. 8 8. 1 8. 1	Grams 15. 5 102 88 81	Percent 12. 00 26. 28 2. 31 8. 83	Percent 98 96 97 94	Grams 2. 5 7. 1 2. 0 16. 8	Per- cent 23. 70 49. 49 5. 41 21. 00

It is seen in the table that the proportion of <sup>20</sup> insoluble organic base to the total base in the peat is 94 percent or higher. When it is considered that arecoline, ethylene diamine, and piperidine are liquids which are completely miscible with water, the efficacy of peat in fixing, or 25 rendering insoluble, organic bases of the types specified, is realized.

Instead of following the above procedure, I may employ a simplified procedure, viz., one in which no separation of the liquid part from the solid 30 is made. The soluble and insoluble compounds of the base are left together and the whole mixture is evaporated to dryness. This mixture contains less of its total base in insoluble form but the percentage is still high enough, the dif-35 ference being only a matter of degree, to make it adaptable for the same uses as the above separated products. This mixed form may be called the "evaporated mixture", or, since it is essentially similar in character to the foregoing products, 40 may also be known by that name—thus, "arecoline peat".

Another procedure which I may employ consists in mixing together the two ingredients, peat and an organic base, without the addition of any water. The resulting mixture is to all outward appearances a dry powder, due to the high liquid absorbing capacity of peat. The base in this dry mixture is in its free or uncombined state, which is readily shown since it can be extracted by an so appropriate organic solvent. The same solvent. on the other hand, will not remove the organic base from a product formed by the reaction between the two constituents in the presence of water. Although this is a mechanical mixture

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in that on the addition of water a reaction occurs, said reaction being to produce substantially the same products as by the aqueous interaction of peat and the organic base.

Having thus defined my invention, what I claim for Letters Patent is:

- 1. The substantially water-insoluble reaction 25 products of peat with organic bases of the group consisting of arecoline, anabasine, codeine, ethylene diamine and piperidine.
- 2. The substantially water-insoluble reaction product of peat with arecoline.
- 3. The substantially water-insoluble reaction product of peat with codeine.
- 4. The substantially water-insoluble reaction product of peat with ethylene diamine.
- 5. The process of preparing substantially wa- 35 ter-insoluble reaction products of peat with organic bases of the group consisting of arecoline, anabasine, codeine, ethylene diamine and piperidine, which comprises bringing together peat and any of said organic bases in the presence of water. 40
- 6. The process of preparing a substantially water-insoluble reaction product of peat and arecoline, which comprises bringing together peat and arecoline in the presence of water.
- 7. The process of preparing a substantially 45 water-insoluble reaction product of peat and codeine, which comprises bringing together peat and codeine in the presence of water.
- 8. The process of preparing a substantially water-insoluble reaction product of peat and 50 ethylene diamine, which comprises bringing together peat and ethylene diamine in the presence of water.

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