PCT WORLD INTELLECTUAL PROPERTY ORGANIZATION
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INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(51) International Patent Classification 5 :		(11) International Publication Number:
C02F 1/28, 1/54	A1	WO 90/05703
		(43) International Publication Date: 31 May 1990 (31.05.90)

(21) International Application Number: PCT/DK89/00263

(22) International Filing Date: 9 November 1989 (09.11.89)

(30) Priority data:
6427/88 17 November 1988 (17.11.88) DK

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(81) Designated States: AT, BE (European patent), CH, DE, DK, FR (European patent), GB, IT (European patent), JP, LU (European patent), NL, SE, SU, US.

Published
With international search report.
In English translation (filed in Danish).

(54) Title: TREATMENT OF SLUDGE

(57) Abstract

Hard swimming layers in drainage conduits for animal sludge manure are decomposed and/or subjected to separation by treatment with a cellulose containing material, preferably straw, which is at least partly decomposed with an aqueous plant and/or wood extract, or are treated with such an extract alone, whereafter the heavy metals or heavy metal compounds, coarser and lighter nutrient-containing particles are separated. The extract is prepared by extraction of preferably finely divided plant and/or wood material in water at a pH of about the neutral point and at a temperature of up to about 30°C. There is obtained an effective separation of solids, including nutrient-containing residual materials which can be reused, as well as heavy metals. Furthermore, the solids can be utilized for the production of biogas.

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Treatment of sludge

The present invention relates to a process for breaking down so-called hard scum deposits and similar materials in liquid manure and sewage ducts and/or for separating the dry matter content from liquid manure, domestic and industrial sludge as well as waste products from crops such as flax stalks.

The invention also relates to plant- and/or wood extract for use, in connection with the process, by way of separation-promoting agent or agent breaking down scum deposits.

Lastly the invention relates to a plant for carrying out the separation process described.

Materials of the above type, in particular liquid manure and various types of sludge, create ever increasing environmental problems, the solution of which has so far gone hand in hand with considerable difficulties, numerous practical disadvantages and, as a result thereof, high costs.

To illustrate the problems and the solutions so far proposed or attempted the treatment of liquid manure, resulting e.g. from the keeping of pigs, shall, in particular, be discussed. The liquid manure is characterised by a large content of water and a small content of dry matter, including nutrient-containing residues and heavy metals or heavy-metal compounds, and it is precisely the composition of the liquid manure, which incidentally varies from concern to concern, which creates considerable problems as regards its treatment, i.e. separation into solid materials of various kinds and water of maximum attainable purity.

With traditional long-time storage of liquid manure in tanks, which is costly to set up, merely a postponement of, not least, the environmental problems is achieved.

It has been proposed to separate liquid manure by chemical treatment with lime. This however merely creates new problems, since the pH value of the material is bound to be very high, typically about 12-12.5. Furthermore, the costs of lime treatment are considerable.

It has been attempted to centrifuge liquid manure in order to separate out the relatively small amount of solid constituents, making use to this end of both traditional and specially designed centrifuges.

However, the results have consistently been unsatisfactory, inasmuch as effective separation has not been achieved.

It is possible to evaporate liquid manure, but this necessitates such considerable amounts of energy as to make the process uneconomic. The transportation of the liquid manure from the various producers to large communal plant with a view to evaporation or some other known treatment may possibly improve the process economy to a certain extent, but the transportation is costly and problematic from the point of view of hygiene, since there is a risk of spreading cattle and pig diseases.

From US patent specification no. 2.117.378 a settling and coagulating agent is known containing in substance all the water-soluble constituents of the sap from plants of the species opuntia cochinellifera, a cactus plant belonging to the family cactaceae. The sap is pressed out of the plant stems and filtered, whereupon it congeals to a sticky, paste-like substance, which is used by way of primary settling agent. The agent can also be produced by dehydration of the plant material in a drying kiln and subsequent pulverisation, and the powder can be purified by dissolving it in water and evaporting it to powder form. If use is made of the congealed type, the material must be protected against decomposition, e.g. by adding to it salicylic acid. The agent can be used for precipitating solid substances from inter alia effluents and sewage.

US patent specification no. 1.369.871 describes a process for converting waste vegetable material to fertiliser by subjecting the vegetable material to the action of flowing waste products from stables.

US patent specification no. 1.890.459 describes a process for producing organic/mineral fertiliser from faecal material contained in effluents or sewage, by mixing the faecal material with 3-5 % by weight of peat or lignite and heating it to about 60°C, whereby the peat or lignite absorbs solid organic fertilising material.

From the Norwegian patent application no. 147.517 published for opposition a process is known for purifying drainage water, in which bark lignin is added to the drainage water, the method in practice being to add NaOH and bark lignin and stir the mixture, whereupon it is acidified with sulphuric acid and the precipitated mass is separated by centrifuging, it being preferable to add aluminium sulphate together with NaOH and bark lignin.

From Japanese patent specification no. 87.051679 a sludge dehydration agent is known, which causes sludge to be efficiently deodorised. The agent contains a polymeric coagulation agent and an oil-type deodorising agent such as wood tar, essential oils or terpenes and surface active agent.

US patent specification no. 1.364.387 describes the use of e.g. calcium carbonate, sand and/or asbestos for precipitating solid matter from sludge and sewage.

US patent specification no. 2.300.693 describes the dehydration of biological waste sludge by adding various metal salts and subsequent filtration.

According to US patent specification no. 3.645.893 solid matter can be precipitated from liquid manure and similar materials by adding e.g. polyacrylic acid, ferric chloride or aluminium sulphate.

The object of the present invention is to provide an economic and in practice efficient process, i.e. a process capable of being carried out easily and quickly, for separating the dry matter contained inter alia in various types of liquid manure and sludge from the very large amounts of water present in these materials, inasmuch as the process is such as to be especially suitable for use at individual places of production, e.g. of individual pig or cattle breeders. With the process according to the invention it is, in addition, possible to break down/dissolve so-called scum deposits and similar materials, which typically form in ducts for liquid manure constituting solid, hard crusts above the flowing material.

The process according to the invention is based on the surprising insight that treatment of e.g. liquid manure with a separation-promoting agent in the form of a plant- and/or wood extract such as described in detail below causes efficient separation into a purified water phase and solid substances, including nutrient-containing residual material and heavy metals or compounds thereof.

The process according to the invention is accordingly unique in that the material to be broken down and/or subjected to separation is treated with a material containing cellulose, preferably straw, at least partly broken down by means of an aqueous plant- and/or wood extract or treated with such an extract alone, whereupon heavy metals or compounds of heavy metals seggregated in the course of the treatment as well as

relatively coarse and relatively light nutrient-containing particles are separated out.

The aqueous liquid left over after separation is advantageously conducted through an organic filter material, preferably a layer of material containing cellulose, in particular straw, partly broken down by means of the extract. However, if as stated below use is made of an extract material based on heracleum mantegazzianum (giant hogweed) filtration is not necessary, and the separated liquid portion can be utilised when producing the extract, using it in connection with a bubbling-through treatment of the extract material.

The plant- and/or wood extract to be used by way of separation-promoting agent with the process according to the invention is unique in that it is produced by extracting, preferably but not necessarily, finely dispersed plant- and/or wood material in water at a pH value close to the neutral point and at a temperature of about 30°C, preferably at about room temperature, whereby in the course of extraction an oxygen-containing gas, preferably air, is preferably bubbled through the aqueous extraction medium.

It has proved that the extract can be produced from an exceedingly wide range of plant- and/or wood materials, which may be in the form of a finely dispersed powder, dust, scrapings, chips, coarsely chopped up root parts etc.

According to the invention it is particularly advantageous to start from a material produced by more or less fine comminution of e.g. spruce, birch, hazel, beech, oak, willow, poplar and/or flax, in particular Douglas fir. It has proved especially advantageous to make use of coarsely chopped up root parts of heracleum mantegazzianum (giant hogweed) possibly combined with straw, in particular flax straw, and especially spruce, birch, hazel, beech, oak, willow, poplar and/or pine.

The extraction can be carried out at temperatures from above freezing point and generally up to about 30°C although in certain cases higher temperature can be applied. The pH value should advantageously be in the region of the neutral point, but can, without disadvantage, also be somewhat higher, e.g. pH = 7.7. Bubbling through with oxygen-containing gas, especially air, is advantageous for achieving reasonably rapid extraction, and by way of a typical example it may be mentioned that if about 1 kg powdered wood from Douglas fir is treated with about

1,000 litres of water for about 3 to 4 hours at about 20°C, bubbling through about 0.3 cbm air per hour, an extract is achieved which is well suited to the objectives of the invention.

By way of a further example it may be mentioned that an effective extract can be produced in the following way:

Pure water is added to straw, and an oxygen-containing gas, preferably air, is bubbled through the material. The air bubbles are applied during a period of about 40 minutes. Once the amount of bubbles corresponds in terms of volume to about half the total amount of material, the extract is effective. With this process the water temperature amounts to about 20°C but it is possible to operate within the range from about 2 to about 30°C.

A more effective extract can be produced by using e.g. 50% straw + 50% flax straw as well as e.g. 3-5% coarsely chopped up root parts of heracleum mantegazzianum.

If use is made of spruce, birch, hazel, beech, oak, willow, poplar and/or pine, a few per cent of chopped up heracleum mantegazzianum have a reinforcing effect on the separation activity of the extract.

The amount of extract to be used for separating e.g. liquid manure of industrial sludge is relatively small but will vary according to the material from which the extract is made, the composition of the liquid manure or sludge and a number of other factors, e.g. the temperature. A number of simple preliminary tests will as a rule indicate what amount of extract exactly is to be added; by way of guidance it can be stated that addition of extract to an amount of more than about 4-5% (vol/vol) up to about 10% (vol/vol) will normally be sufficient in order to achieve effective separation. In practice an amount from about 6 to about 10% (vol/vol), especially from about 6 to about 8% (vol/vol) will often be advantageous; the amount is not critical since in general the position is that a small amount of extract merely necessitates a longer separation time than a larger amount.

The process here described can be carried out in every type of suitable apparatus or plant but it has proved advantageous, especially when separating liquid manure, to make use of a plant unique in that it comprises a main vessel with built-in heating devices and an upper

inlet for the material to be separated, e.g. liquid manure or sludge, as well as an outlet for purified water phase, an upper vessel arranged above the main vessel and connected with the latter with a gas outlet and with devices for the removal of separated light material such as a screw conveyer, a first lower vessel containing cooling devices and devices for the removal of solid matter arranged below the main vessel and connected with the latter, and a further lower vessel connected with the first-named lower vessel and containing devices for the removal of solid matter.

In practice the plant is applied in such a way that the extract, possibly together with thereby partly broken down cellulose-containing material, preferably straw, is fed, within the main vessel, which if required is heated, into the liquid manure or sludge. After a separation time, which typically amounts to about 6-8 hours, the constituents of the material will be separated; heavy metals and compounds thereof, typically zinc and cadmium or compounds thereof, will be precipitated into the lowest lower vessel, from where it can be removed for stockpiling or possible recovery of the metals. The relatively coarse particles will be precipitated into the upper (first) of the lower vessels, whereas the lighter particles rise upward and are collected in the upper vessel where they form a porous, spongy mass. Thereafter the main vessel contains water with a small amount of dry matter which can be returned to the liquid manure duct, if required via a vessel containing straw or similar matter partly broken down by means of extract, which acts as a filter for the finest dry matter particles and absorbs a part of the water. The straw can later be used for degassing. In the course of the separation process gas is developed, principally methane, which is removed from the gas outlet of the upper vessel for possible collection in later use.

The porous, spongy mass which collects in the upper vessel is picked up with the aid of e.g. a built-in conveyer screw and can be used for degassing. The coarser material separated in the upper one of the lower vessels can be easily centrifuged and is also used for degassing. The solid residual substances accordingly present after degassing have, depending on the type of material treated (liquid manure, sludge etc.) a not insignificant content of nutrient substances, which if desired can be made use of in dry fertiliser mixtures or

possibly feeding stuff mixtures, whereby the overall result is not only cheap and efficient separation of liquid manure, sludge or similar materials but also the possibility of reusing the solid constituents and of producing biogas, in particular methane.

With a view to degassing the dry matter, which after the separation process may be present in a concentration of 20-80%, it is advantageous to move the dry matter together with a small amount of water gently by an up-and-down movement, a kind of kneading, which may be done in any rockable vessel.

According to the invention liquid manure from small pigs weighing 5-25 kg and the fodder utilisation of which is very small can be separated without adding broken down straw or similar material. The separation can e.g. be brought about by adding 20% (vol/vol) extract made from e.g. flax straw and poplar.

In a perforated module arranged in the lowest part of the separation vessel, which contains a fibrous material consisting of flax straw and poplar, an oxygen-containing gas, preferably air, is bubbled through.

Introduction of the liquid manure into the module and the effect of the added extract produce rapid, coagulation-like separation.

The water portion can, by addition of e.g. 6-10% (vol/vol) extract be returned to the liquid manure duct, where it will initiate a weak separation in the flowing liquid manure formed.

With an amount of liquid manure of about 1,000 litres a very fine separated consistency of dry matter is achieved in the course of about 2-3 hours at about 16-20°C if altogether about 0.6 cbm air is bubbled through, whereby the high nutrient substance content of the dry matter makes it well suited for further use.

According to the invention the extract described, possibly cellulose-containing material partly broken down thereby, can be used for breaking down (dissolving) so-called hard scum deposits which are prone to collect in liquid manure ducts and similar locations. Such scum deposits are thick, rigid and especially hard, and they can prevent emptying of the ducts. If the extract material is added periodically, the scum deposit is quickly dissolved or broken down to a mud-like consistency, in which process it sinks to the bottom of the duct, whereby a small part or none of the liquid manure's dry matter

will be at the surface. This causes the amount of hydrogen sulphide given off to be reduced considerably. The liquid manure is pumped to a separation plant. It can for instance be noted that in a transverse duct for collecting liquid manure from a liquid manure duct containing a rigid and undesirable scum deposit altogether about 40 litres of extract (pH = about 7.2) was added, over a period of 2 days, to altogether about 60 cbm of thick liquid manure. The rigid and hard scum deposit was gradually broken down, and after return-pumping for a short period of time the liquid manure had become a homogenous mass, which later did not lead to the formation of a scum deposit.

By way of further illustration of the invention the following example can be cited:

At the enterprise of a pig breeder with a stock of 350 sows and additionally 2,400 porkers the daily amount of liquid manure was 27 tons, and the liquid manure storage capacity about 6 months.

The liquid manure could not be pumped, since the liquid manure ducts had not been emptied effectively for about $3\frac{1}{2}$ years.

An extract according to the invention, based on about 15 kg flax straw (not chopped up) and 0.75 kg coarsely chopped up root parts of heracleum mantegazzianum and water and produced by the above bubbling-through method was after about 2 hours bubbling through introduced into the liquid manure ducts in an amount of about 1% (vol/vol).

Virtually at once there were visible signs of the liquid manure being homogenised, the scum deposit disappeared and so in part did the undesirable smells. 48 hours after addition the ducts could be emptied totally, for the first time in $3\frac{1}{2}$ years.

If use was made of 5-6% (vol/vol) of the extract, the dry matter was precipitated within 5-6 hours; however, this must be done outside the liquid manure ducts, since the dry matter portion cannot be pumped.

The ducts were kept free for addition of 1% (vol/vol) extract once every week.

After about one week the separated solid material was spread in the field without any unpleasant smell.

An extract of the type described above has, in practical tests on poultry muck (2 tons) produced the same results as regards the

homogenisation and separation of liquid and dry matter.

The principle of the present invention can in practice also be applied for breaking down e.g. such residual products as straw and/or stalks from grain, flax and rice crops and other vegetable crops, in particular linseed. For the extract described has proved to be effective for breaking down these residual products in such a way that after treatment in a mixer they can be converted to a pulp suitable for gas production or to a paste which can be used as a soil-improving agent.

It should also be noted that e.g. compost from domestic waste and certain types of industrial waste, which frequently contains rather large amounts of heavy metals or compounds thereof, can be treated by the prccess according to the invention for removal of the undesirable metal components. The separated substances not ccntaining metals can be used for gas production and/or be, after centrifuging, reused for fertilising purposes.

The plant and/or wood extract here described will of course vary in composition, depending on its origin. It is assumed, without the invention being in any way limited thereby, that the extracts probably owe their effectiveness in every case partly to their presumed content of various types of terpene, break-down products thereof and/or hydrolysis and oxidation products therecf.

C L A I M S :

1. Process for breaking down so-called hard scum deposits and similar materials in liquid manure and other sewage ducts and/or for separating the dry matter content from liquid manure, domestic and industrial sludge as well as waste products from crops such as flax stalks, c h a r a c t e r i s e d in that the material to be broken down and/or subjected to separation is treated with a cellulose-containing material, preferably straw at least partly broken down by an aqueous plant- and/or wood extract or treated with such an extract alone, whereupon one separates heavy metals or compounds of heavy metals seggregated during treatment as well as relatively coarse and relatively light nutrient-containing particles.

2. Process according to claim 1, c h a r a c t e r i s e d in that the aqueous liquid remaining after separation is conducted through an organic filter material, preferably a layer of cellulose-containing material, in particular straw, partly broken down by the extract.

3. Process according to claim 1 or 2, c h a r a c t e r i s e d in that the separated particles are made use of in a degassification process.

4. Plant- and/or wood extract for use in the process according to each of the preceding claims, c h a r a c t e r i s e d in that it is produced by extraction of preferably finely dispersed plant- and/or wood material in water, with a pH value in the region cf the neutral point and at a temperature up to about 30°C, preferably at about room temperature, in that during the extraction an oxygen-containing gas, preferably air, is bubbled through the aqueous extraction medium.

5. Extract according to claim 4, c h a r a c t e r i s e d in that it is produced from spruce, birch, hazel, beech, oak, willow, poplar and/or flax, and preferably from Douglas fir.

6. Extract according to claim 4, c h a r a c t e r i s e d in that it is produced from root parts of heracleum mantegazzianum.

7. Plant for carrying out the process according to each of claims 1-3, c h a r a c t e r i s e d in that it comprises a main vessel with built-in heating devices and an upper inlet for the material to be separated, e.g. liquid manure or sludge, as well as an outlet for purified water phase, an upper vessel arranged above the main vessel and connected with the latter, said upper vessel having a gas outlet and devices for the removal of separated light material as well as a screw conveyer, a first lower vessel arranged below the main vessel and connected with the latter, said first lower vessel containing cooling devices and devices for the removal of solid matter, as well as a further lower vessel connected with the first-mentioned lower vessel and containing devices for the removal of solid matter.

INTERNATIONAL SEARCH REPORT

International Application No PCT/DK 89/00263

I. CLASSIFICATION OF SUBJECT MATTER (if several classification symbols apply, indicate all) [6]

According to International Patent Classification (IPC) or to both National Classification and IPC

IPC5: C 02 F 1/28, 1/54

II. FIELDS SEARCHED

Minimum Documentation Searched [7]

Classification System	Classification Symbols
IPC5	A 01 C; B 01 D; C 02 F; E 03 F

Documentation Searched other than Minimum Documentation to the Extent that such Documents are Included in the Fields Searched [8]

SE,DK,FI,NO classes as above

III. DOCUMENTS CONSIDERED TO BE RELEVANT [9]

Category *	Citation of Document, [11] with indication, where appropriate, of the relevant passages [12]	Relevant to Claim No. [13]
X	US, A, 2117378 (GEORGE E. TIFFANY) 17 May 1938, see the whole document --	1-5
X	US, A, 1369871 (ALBERT C. ZIRWAS ET AL) 1 March 1921, see the whole document --	1
X	US, A, 1890459 (RICHARD FLUCK ET AL) 13 December 1932, see the whole document --	1
X	GB, B, 532877 (AFRICAN SISAL & PRODUCE CO., LTD.) 24 April 1941, see the whole document --	1,4

* Special categories of cited documents: [10]

"A" document defining the general state of the art which is not considered to be of particular relevance

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"O" document referring to an oral disclosure, use, exhibition or other means

"P" document published prior to the international filing date but later than the priority date claimed

"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step

"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.

"&" document member of the same patent family

IV. CERTIFICATION

Date of the Actual Completion of the International Search	Date of Mailing of this International Search Report
16th February 1990	1990 -02- 26
International Searching Authority	**Signature of Authorized Officer**
SWEDISH PATENT OFFICE	Bo Bergström

III. DOCUMENTS CONSIDERED TO BE RELEVANT (CONTINUED FROM THE SECOND SHEET)

Category *	Citation of Document, with indication, where appropriate, of the relevant passages	Relevant to Claim No
X	GB, B, 544810 (C.L. WALSH ET AL) 23 July 1942, see the whole document --	1,4
X	DE, C, 649188 (HEINZ DICKMANN) 17 August 1937, see the whole document --	1
X	Derwent's abstract, No. 86- 5 561/01, SU 1 162 454, publ. week 8601 --	1
X	US, A, 1364387 (CLARENCE P. LANDRETH) 4 January 1921, see the whole document --	7
X	US, A, 2160832 (PETER M. CONTANT) 6 June 1939, see the whole document --	7

FURTHER INFORMATION CONTINUED FROM THE SECOND SHEET

V. ☐ OBSERVATIONS WHERE CERTAIN CLAIMS WERE FOUND UNSEARCHABLE [1]

This international search report has not been established in respect of certain claims under Article 17(2) (a) for the following reasons:

1. ☐ Claim numbers....., because they relate to subject matter not required to be searched by this Authority, namely:

2. ☒ Claim numbers....., because they relate to parts of the international application that do not comply with the prescribed requirements to such an extent that no meaningful international search can be carried out, specifically:

Claims incompletely searched: 1-3 and 4-5, because the starting materials for the extracts embrace too many different kinds of plants and trees that give extracts with too many different properties.

3. ☐ Claim numbers....., because they are dependent claims and are not drafted in accordance with the second and third sentences of PCT Rule 6.4(a).

VI. ☐ OBSERVATIONS WHERE UNITY OF INVENTION IS LACKING [2]

This International Searching Authority found multiple inventions in this international application as follows:

1. ☐ As all required additional search fees were timely paid by the applicant, this international search report covers all searchable claims of the international application.

2. ☐ As only some of the required additional search fees were timely paid by the applicant, this international search report covers only those claims of the international application for which fees were paid, specifically claims:

3. ☐ No required additional search fees were timely paid by the applicant. Consequently, this international search report is restricted to the invention first mentioned in the claims; it is covered by claim numbers:

4. ☐ As all searchable claims could be searched without effort justifying an additional fee, the International Searching Authority did not invite payment of any additional fee.

Remark on Protest

☐ The additional search fees were accompanied by applicant's protest.

☐ No protest accompanied the payment of additional search fees.

ANNEX TO THE INTERNATIONAL SEARCH REPORT ON INTERNATIONAL PATENT APPLICATION NO. PCT/DK 89/00263

This annex lists the patent family members relating to the patent documents cited in the above-mentioned international search report.

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
US-A- 2117378	17/05/38	NONE	
US-A- 1369871	01/03/21	NONE	
US-A- 1890459	13/12/32	NONE	
GB-B- 532877	24/04/41	NONE	
GB-B- 544810	23/07/42	NONE	
DE-C- 649188	17/08/37	NONE	
US-A- 1364387	04/01/21	NONE	
US-A- 2160832	06/06/39	NONE	