The invention is directed to a process for the treatment of manure comprising treating the manure to separate the solids from the manure, thereby obtaining at least one liquid fraction and a solids fraction, treating the said at least one liquid fraction by reverse osmosis to obtain a pure water fraction and a retentate, freeze crystallizing the retentate to obtain an ice fraction and a concentrated manure fraction, combining the said concentrated manure fraction with the said solids fraction to obtain a fertilizer product and optionally drying the said fertilizer product, melting the said ice fraction and treating the resulting liquid to reverse osmosis.
Title: Process for the treatment of manure

The invention is directed to a process for the treatment of manure.

The large surplus of pig manure in Europe requires the development of different technologies for the economical processing and reuse of this by-product of the meat industry. Some of these systems produce a concentrated liquid stream that can be used as natural fertilizer at arable lands. However this arable land is often not located at the pig farm itself since this type of farming does not require any grasslands. Therefore the liquid fractions must be stored and transported to regions where it can be applied as fertilizer. The high costs associated with the storage and transport of these liquid fractions make it interesting to look for methods of reducing the volume of these streams.

An existing process is shown in figure 1, wherein raw manure is separated into a liquid and a solid fraction. The solid fraction is stored (after eventual drying) and can be used for soil improvement (it is a phosphorous rich product), incinerated or processed otherwise.

The liquid fraction is treated in an air flotation unit to remove remaining solids, which are recycled to the initial liquid/solid separation. The resulting liquid fraction is concentrated in a reverse osmosis unit to produce clean water and a mineral concentrate.

As indicated above a disadvantage of this process is the relative large volume of the mineral concentrate which makes it uneconomic to use the concentrate as fertilizer, due to the high transport costs involved.

Accordingly it is an object of the present invention to overcome this disadvantage. Further objects will become apparent from the description of the invention.

The invention is directed to a process for the treatment of manure, more in particular pig manure, comprising treating the manure to
separate the solids from the manure, thereby obtaining at least one liquid fraction and a solids fraction, treating the said at least one liquid fraction by reverse osmosis to obtain a pure water fraction and a retentate, freeze crystallizing the retentate to obtain an ice fraction and a concentrated manure fraction, combining the said concentrated manure fraction with the said solids fraction to obtain a fertilizer product and optionally drying the said fertilizer product, melting the said ice fraction and treating the resulting liquid to reverse osmosis.

An important advantage of the process according to the invention is that less mineral scaling and little to no odour emissions arise during the process due to the low operating temperatures. The latter is an important benefit when applying the process in more densely populated areas. In addition the energy consumption for freeze concentration is significantly lower than for commonly used evaporative systems.

In a first embodiment of the present invention it is preferred to treat the reverse osmosis retentate by freeze crystallization under such conditions that only two fractions are obtained, namely a (substantially solids free) concentrated manure fraction and an ice fraction. This means that the conditions of the freeze crystallization are such that the temperature is kept above the eutectic freezing point of the system. In this way no separate solid salt fraction is formed. The advantage thereof is that it is not necessary to treat this salt fraction separately. The actually preferred temperature for the freeze crystallization step is below 0°C and above the eutectic freezing point of the fraction that is treated. This temperature will depend on the composition of the fraction and the amount of volume reduction required (i.e. the amount of ice to be produced).

The ice fraction will be slightly contaminated by adhering concentrated manure. The ice fraction is molten and treated in a reverse osmosis (RO) step, either the step for the original liquid fraction, or a separate RO-unit, specifically designed for this composition. The latter
possibility is also useful in case of redesigning or extending an existing manure treatment unit, already having an RO-step, which may be too small to treat the total liquid volume.

Heat exchange may be advantageously applied between the melting step and the freeze crystallization step.

According to another embodiment the reverse osmosis retentate may be treated in a eutectic freeze crystallization step, resulting in three fractions, namely an ice fraction, a concentrated manure fraction containing solid salt particles (as a third fraction). The concentrated manure containing the solid salt may be separately treated to obtain a solid or semi-solid salt fraction.

According to the invention the concentrated manure (brine) (optionally in combination with the solid salt (if produced)) is combined with the initially obtained solid fraction. The combined product can be used as such, for example as wet fertilizer, or dried to a solid product.

In this way the manure is converted to clean water and a solid product that can easily and economically be transported, without any further waste streams (Zero liquid discharge). The process surprisingly provides for a very economic and easy process to treat manure and to convert it to useful components, substantially without any loss in material, or waste product.

**Detailed description of the invention**

In the first step of the process of the present invention, the manure is split into a liquid and a solids fraction. This step uses conventional separation techniques known in the art of manure treatment. In general the said first step comprises two separate sub-steps, in the first one the rough solids are removed, following which in the second step the remaining finer, suspended solids are removed, for example by air flotation.
These finer solids are recycled to the first step and combined therein with the first rough solids fraction. Examples of methods for the separation of the rough solids are belt filters, filter presses, etc..

The solids fraction is preferably used as such as fertilizer, optionally after drying, after combining with a stream obtained after downstream treatment.

The liquid fraction is treated in a reverse osmosis unit. This unit produces on the one hand clean water, and on the other hand a concentrated mineral fraction as retentate. However, this fraction contains mainly water, which is very uneconomical to transport. According to the invention, the retentate is further treated by freeze crystallization. By the use of this step the retentate is further concentrated.

It has been found that freeze crystallization is a very suitable method for this, resulting in relatively low energy consumption (compared to evaporation based alternatives), little issues with smell and corrosion due to the low temperatures, electricity is used as energy input (i.e. no fuel needed for heaters), no high/low pressures required and no hot parts are present leading to a relatively safe process environment. The ice produced is, after melting, further be treated in a reverse osmosis unit to remove the final contaminants. This reverse osmosis unit is preferably the same unit as used upstream of the freeze crystallization.

According to the invention the concentrated manure fraction, obtained in the freeze crystallization step is combined with the first solids fraction, which, after optional drying, is a suitable fertilizing material. As discussed above, in one embodiment the freeze crystallization step is preferably done at a temperature well above the eutectic freezing point of the mixture, thereby producing only an ice fraction and a concentrated manure fraction.

The melting of the ice is preferably done in an integrated manner with the remainder of the process, for example by using the ice to pre-cool
one or more feed streams to the freeze crystallizers, or to condense refrigerant for the cooling machines to be used in the process. In this way both the sensible heat and the melting energy is used efficiently.

The permeate of the RO unit is a clean water product which may be used in the process, optionally after further removing the final contaminants by ion exchange.

In another embodiment, the freeze crystallization (or concentration) is done under eutectic conditions, resulting in ice and a concentrated manure fraction containing a part of the minerals in a crystallized form. Eutectic freeze crystallization has, in this embodiment the advantage that higher volume reductions can be achieved. This means that less liquid is added to the solid product (thus reducing the required drying duty).

This aspect is based on the use of eutectic freeze crystallisation, which is a process based on separation of components at a eutectic freezing point. Eutectic freeze crystallisation has been described in EP-A 1,230,194 and in Chem.Eng.Proc. 37, (1998), pp 207-213.

With the use of the eutectic freeze crystallization in the process of the present invention, the process can become a ‘zero waste’ process, meaning that the manure is completely converted to useful or harmless products.

The process of the invention is now further explained on the basis of the figures, wherein in figure 1 a process according to the prior art is described, in figure 2 one embodiment of the invention is described and in figure 3 another, more preferred embodiment of the invention is described.

In figure 1 raw pig manure is fed from a storage 1 to separation unit 2, wherein the manure is separated into a liquid fraction and a solids fraction. The liquid fraction is transported to a dissolved air flotation unit 3. In this unit the remaining solids are substantially removed from the liquid fraction and returned to the separation unit 2. From this unit 2, a solid
product is recovered for further use, optionally after drying (not shown), or discharged.

The liquid fraction from the flotation unit 3 is subjected to reverse osmosis in RO unit 4, to produce clean water and a mineral concentrate as retentate.

In figure 2 a general description of the process of the invention has been shown. This process differs from the process of figure 1 in that it the mineral concentrate from the RO (unit 4) is subjected to freeze concentration in 5 whereby the molten ice product (which contained some adhering mother liquor) is recycled to the RO unit for purification. The permeate from the RO is a clean water product. The concentrate product from the freeze concentration can be used as a mineral concentrate for fertilizing purposes. The solids product of the separation unit 1 is dried in drier 6.

In figure 3 the mineral concentrate is combined with the solids fraction from separation unit 2 and subsequently dried to produce a solid product that can be used as fertilizer (mineral enriched).

The invention is now elucidated on the basis of the following, non-limiting example.

EXAMPLE

A manure stream containing 8 wt% suspended solids (TSS) and a concentration of dissolved compounds (TDS) of 2.2 wt% is fed to a belt filter to remove the solids as a wet solid product (TSS > 30%). The liquid stream leaving the belt filter enters a dissolved air flotation unit (DAF) to remove the remaining solid particles, the latter are recycled back to the belt filter. The now solid free liquid stream (together with a further stream to be discussed later) is the feed to a reverse osmosis (RO) unit where it will be concentrated to a typical value for TDS of 5 wt%. The permeate is the clean
water product and can be used as process water at the pig farm, discharged in the environment or other.

The RO retentate is fed to a freeze crystallizer where it is partly converted into ice. The freeze crystallization can be either performed in one or in multiple crystallizers placed in series, depending on the type of equipment used and the desired final concentration level. This example shows a lay-out for a serial set-up.

The RO concentrate is pre-cooled to a temperature of -1°C before entering the first crystallizer. This crystallizer operates at a constant temperature of about -3.5 °C. This will convert 25% of the incoming mass into ice. The ice/concentrate mixture is separated by gravity in a specially selected settler. It is expected that around 10% of the produced ice will melt in this stage. A ice slurry stream with a TSS of 70 wt% is extracted from the settler. The concentrate (TDS = 6.8 wt%) is the feed for the next crystallizer which is operating at a temperature of around -5°C. A similar arrangement as the one described above result in an ice slurry and a concentrate with a TDS of 9.4 wt%. Again this concentrate is further concentrated in a third crystallizer (operating temperature of -6.5 °C) yielding another ice slurry and the final concentrate with TDS of 13 wt%. The final concentrate is combined with the wet solid product coming from the belt filter and dried.

The three ice slurry streams are combined and molten in a special heat exchanger. Both the sensible as and the latent heat of the ice stream can be utilized for heat integration purposes, e.g. pre-cooling the feed to the first crystallizer and condensation of the refrigerant in the cooling machine at a low temperature (this leads to a significant reduction in the electricity demand of the compressor). The now molten ice stream (TDS = 2.6 wt%) is combined with the DAF effluent and this combined stream (TDS = 2.4 wt%) is thus the feed to the RO-unit.
The process in this example results in a conversion of the raw manure into clean water and a dry (TSS >80%) solid product accounting for 64 wt% and 12 wt% of the incoming mass, respectively.
Conclusies

1. Werkwijze voor het behandelen van mest, omvattende het behandelen van de mest om de vaste stof van de mest te scheiden, waarbij ten minste één vloeibare fractie en ten minste één vaste stof fractie verkregen wordt, het behandelen van de ten minste één vloeibare fractie met behulp van omgekeerde osmose voor het verkrijgen van een fractie zuiver water en een retentaat, het vries-kristalliseren van het retentaat voor het verkrijgen van een ijs-fractie en een geconcentreerde mestfractie, het combineren van genoemde geconcentreerde mestfractie met genoemde vaste stof fractie voor het verkrijgen van een mest product en eventueel het drogen van genoemd mest product, het smelten van de ijs fractie en het behandelen van de verkregen vloeistof met behulp van omgekeerde osmose.

2. Werkwijze volgens conclusie 1, waarbij de scheiding van vaste deeltjes uit de mest twee stappen omvat, waarbij de eerste stap de scheiding omvat van grove vaste deeltjes, en de tweede stap de scheiding is van in suspensie aanwezige vaste stoffen door middel van flotatie.

3. Werkwijze volgens conclusie 1 of 2, waarbij de step van het vrieskristalliseren slechts twee stromen produceert, namelijk de ijsfractie en de geconcentreerde mest fractie.

4. Werkwijze volgens conclusie 1 of 2, waarbij de vries kristallisatie uitgevoerd wordt op het eutectische vriespunt van de voeding aan de vries kristallisatie stap.

5. Werkwijze volgens conclusie 1-4, waarbij genoemde ijsfractie tenminste 85 gew.% ijs bevat en verder moederloog.

6. Werkwijze volgens conclusie 5, waarbij d gesmolten ijsfractie toegevoerd wordt aan dezelfde omgekeerde osmose stap als genoemd tenminste een vloeibare fractie.