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(71) Applicant: **TE Consulting House 4 Plus, SL**
47003 Valladolid (ES)

(72) Inventor: **GONZÁLEZ CALVO, Rafael**
Valladolid (ES)

(54) **METHOD AND FACILITY FOR STATIONARY THERMAL HYDROLYSIS OF ORGANIC MATERIAL WITH TOTAL ENERGY RECOVERY**

(57) Procedure for the thermal hydrolysis of organic matter in steady state, with a double steam explosion and total energy recovery, which consists, as a minimum, of the 1) feeding stage, stepped pressurization and sequential injection of low, medium and high pressure level steam; 2) first stage of hydrolysis by consecutive steam explosion operations with the production of medium pressure level steam and thermal reaction; 3) second stage of hydrolysis consisting of steam explosion and production of low pressure steam. An installation for the implementation of the process, which consists of comprising pumps for stepped pressurization, fluid-steam mixers, valves, mixers, decompression elements, tanks, piping and instrumentation and control systems.

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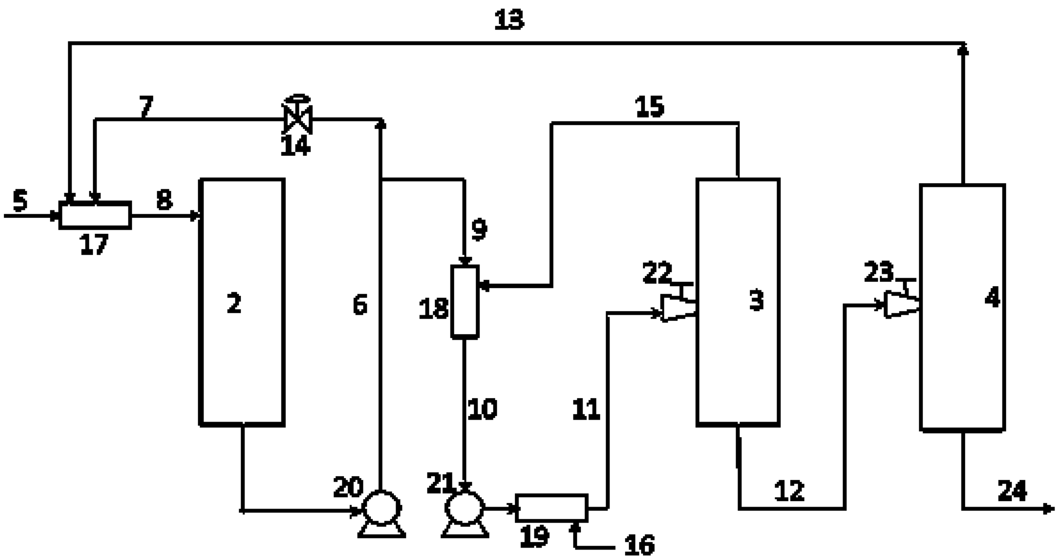

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Description

Technical sector of the invention

[0001] The invention, which includes a procedure and the installation for its implementation, is applied to the field of organic matter treatment, including sewage sludge from wastewater, domestic and industrial waste and any other matter. It finds a special application as a pre-treatment to the anaerobic digestion of solids.

Background of the Invention

[0002] In different industrial processes it is necessary to modify the physical and chemical structure of the solids, for which it's necessary to implement a pre-treatment. In the particular case of anaerobic digestion of solids, the hydrolysis stage (solubilization, liquefaction) limits the overall kinetics of the process. To improve the kinetics of the hydrolysis stage, different physical, chemical and biological processes are applied as a previous treatment to anaerobic digestion, with the consequent improvement of the overall methanization process. The process of thermal hydrolysis is based on subjecting the solid to high temperatures and pressures for relatively long periods of time, normally greater than 30 minutes. Subsequently, taking advantage of the high pressure, the hot mass can undergo a process of sudden decompression or flash process, in order to achieve the so-called steam explosion effect that generates a breakage in the solids structure. Other processes use heat exchangers to recover the energy of the hot mass.

[0003] All technologies that recover energy by recirculating and condensing the steam produced in the flash stage to heat the feed that reaches the hydrolysis stage, use steam of a single pressure level. As this pressure corresponds to that of the flash chamber, the steam produced is low pressure. With this configuration it is not possible to take advantage of all the energy contained in the steam, which leads to energy inefficient systems. In order to reduce energy consumption, it is necessary to concentrate the feed, up to very high values, with the consequent increase in the costs attributable to this operation, which requires the use of high quantities of chemical products. The patent ES2570812A1, aims to solve the inefficiency by raising the pressure of the steam that leaves the flash chamber using a mechanical compressor, which uses electrical energy and has a complex maintenance, by compressing steam containing condensable and incondensable aggressive products; also, when using electrical energy, the system is energetically inefficient.

[0004] Patent ES2538176B1 resolves said energy inefficiency with a radically different conceptual design, which differentiates it from all existing processes and installations. Instead of using a single pressure level steam, it uses steam with two pressure levels. With this configuration and in accordance with the laws of thermodynamics it is possible to recover the energy content of the steam, achieving that the facilities are energetically self-sufficient even operating with moderate concentrations of solids. Starting from this fact, all the procedures and installations that operate with steam explosion use a sequence of thermal reaction stage followed by steam explosion. The claimed process uses a sequence of steam explosion stages + thermal reaction stage + steam explosion stage. This procedure improves the overall kinetics of the process, which leads to more compact installations and lower installation, operation and maintenance costs.

[0005] The present patent derives and is a continuation of the patent ES2538176B1, expanding its field of action. The evolution of the pumping equipment and the appearance of new materials allow, at present, to use mechanical impulsion systems on the fluid to be hydrolyzed which are reliable, low maintenance and capable of supporting the operating conditions. In view of this new situation, the present patent, maintaining the fundamental characteristic of operating with total energy recovery, with low residence times and a sequence of stages of steam explosion + thermal reaction + steam explosion, introduces the possibility of using mechanical pumping systems.

Explanation of the invention

[0006] The present process for the continuous thermal hydrolysis of organic matter consists, as a minimum, of the stages: 1) feeding, stepped pressurization and sequential injection of low, medium and high-pressure level steam; 2) first hydrolysis stage by consecutive operations of steam explosion with production of medium pressure level steam and thermal reaction; 3) second hydrolysis stage consisting of steam explosion and production of low-pressure steam.

[0007] In the usual practice of sludge-generating plants, the inlet temperature to such process is that of the environment, however, there is a tendency to take advantage of the energy of surplus streams. This implies a possible wide range of entry temperatures than that of stage 1, which requires a flexible conceptual design of the procedure and the installations.

[0008] In stage 1, the organic material to be hydrolyzed (5) is pressurized and heated until reaching the setpoint values prior to its entry (11) to stage 2. Pressurization is achieved, at least, by the mechanical impulsion equipment for fluids (20) and (21). The heating is achieved by the injection of at least low (13), medium (15) and high (16) pressure steams. To achieve an adequate quality of fluid-vapor mixture, at least mixers (17), (18) (19) are used.

[0009] The mechanical impulsion equipment for the fluids (20) and (21) and any other to be installed are selected among any type of pumps capable of fluids impulsion, including non-Newtonian ones, and high viscosity suspensions. Particularly in a variant of the invention and thanks to the staggering of pressures, centrifugal pumps can be used, which are cheaper and lower in maintenance costs than positive displacement pumps used by other technologies. They must supply pressures between 1 and 10 barg and operate in a temperature range between 20 - 180 °C.

[0010] The mixers (17), (18), (19), are selected between both static and dynamic equipment.

[0011] Taking into account the great variability of possible types of organic matter and the pressure, temperature and concentration conditions of the feed (5), the procedure and the installation must be flexible. In a variant of the invention, the output stream (6) of the pump (20) is divided into the streams (7) and (9). The stream (7) after passage through the mixer (17) is returned to the tank (2), so that the pump (20) acts simultaneously as pressurization and recirculation system. In another variant, by closing the valve (14) the output stream (6) of the pump (20) is brought directly to the mixer (18), thus breaking the recirculation loop. The feed (5) and the low steam (13) are conducted directly to the tank (2).

[0012] In a variant of the invention, the mixer (17) is an ejector that uses the mass recirculated by the pump (20) as the impulsion fluid. This way, in addition to achieving a good mixing, it is possible to lower the pressure of the chamber (4) and the temperature of the output stream (24). This fact leads to a lower consumption of energy in the process.

[0013] According to the energy balances, the fluid (10) cannot reach temperatures higher than 170 °C, which the literature marks as the limit for the development of secondary reactions that lead to the formation of recalcitrant, non-biodegradable and potentially toxic compounds. In the mixer (19) the extremely rapid mixture of live steam (16) and fluid to be hydrolyzed (10) is achieved, reaching temperatures that can reach up to 220 °C. The heating is carried out in extremely short periods of times, less than 5 seconds, so that said secondary reactions are prevented.

[0014] The fluid (11) at high temperature and pressure passes through the element (22) able to cause instantaneous decompression, with the consequent temperature drop, generating a first breakage in the structure of the solid by the steam explosion mechanism. In the tank (3), a medium pressure steam flow (15) is produced which is conducted to stage 1. The liquid fraction that has suffered a first steam explosion is kept inside the tank (3) between 1 and 15 minutes, thus allowing the temperature hydrolysis reaction process to take place. The pre-hydrolyzed organic matter stream (12) is conducted to stage 3.

[0015] In a variant which is operated with sewage sludge without prior treatment, the tank (3) is controlled so that the temperature of the liquid inside can never exceed 170 °C. In another variant operated with previously digested sludge or with organic matter from another source, the temperature can rise to higher values, always below the secondary reactions appearance limit.

[0016] The fluid (12) whose pressure is that of equilibrium with the temperature imposed as a reference to the tank (3) is depressurized when crossing the element (23). In the tank (4) the low-pressure steam stream (13), which is carried to stage 1 and the stream of hydrolyzed organic matter (24) that is brought to digestion are separated.

[0017] In a variant of the invention, the tank pressure (4) is slightly higher than the atmospheric pressure, in the range between 0-0.2 barg. In another variant and due to the effect of the mixer (17), which in this variant is an ejector acting as a vacuum system, the pressure in the tank (4) can be sustained between -0.5 and 0 barg.

[0018] As a consequence of the procedure used, the system not only operates continuously, but unlike existing technologies, it also operates in a steady state. This means that once the operating variables have been prefixed (e.g. flows, levels, pressures and temperatures), they do not change over time. This behavior leads to processes that operate with greater stability, require simpler control systems, are more flexible and robust, and obtain better performance.

Brief description of the figures

[0019] Figure 1 shows a diagram of the installation for the implementation according to the invention.

Explanation of an embodiment

[0020] Following Figure 1 the procedure is described according to the invention claimed and the means used to perform an installation.

[0021] The means used are: tanks or deposits (2), (3), (4); mechanical impulsion and fluid pressurization equipment (20), (21); fluid-steam mixers (17), (18), (19); expansion elements (22), (23).

[0022] In the selected variant, sewage sludge is hydrolyzed, previously concentrated and at room temperature (5). In the ejector (17), the feed mixture (5) is produced, with low pressure steam (13) and with the recirculation stream (7). The output stream (8) of the ejector is returned to the deposits (2), in order to close a recirculation loop. All low steam (13) condenses in the system. Depending on the design conditions and the recirculation flow rates imposed, the stream (9) exiting the recirculation loop has a temperature between 70 and 100 °C and a pressure with values between 3 and 8 barg.

[0023] The stream (9) after the first pressurization and heating stage receives the medium pressure vapor (15) in the mixer (18). According to the energy balances, in the operating conditions the temperature after the incorporation of medium and high-pressure steam streams moves between 120 and 160 °C, without reaching the thermal level corresponding to the development of secondary reactions. This fluid (10) is pressurized by the pump (21), reaching pressures between 8 and 20 barg. In the ultra-fast mixer (19) the fluid receives a live steam injection (16) with a pressure between 10 and 22 barg, capable of raising its temperature to 160-220 °C.

[0024] After this ultra-rapid heating, the high temperature and pressure sludge (11) passes through the decompression element (22) and splits in the tank (3) in a medium pressure steam stream (15) which is conducted to the stage 1 and in a liquid stream (12). The liquid is kept in the tank (3), during a predetermined time, which varies between 1 and 15 minutes. With this sequence the organic matter is first subjected to a steam explosion process and then to a reaction process by thermal process. Consequently, the tank (3) fulfils the double function of flash tank and reactor. In the case described, the temperature inside the tank (3) is maintained at a pre-set and controlled setpoint value between 140 and 180 °C.

[0025] After remaining in the tank (3) during the preset reaction time, the pre-hydrolyzed organic material stream (12), with a pressure between 3 and 9 barg, passes through the decompression element (23) and into the tank (4), which acts as a flash chamber, splits in a low pressure steam stream (13) that is carried to stage 1 and in a stream (24) of hydrolyzed sludge that is brought to digestion. The tank pressure (4) can vary between -0.5 and 0.5 barg. In the variant described and with the ratio of liquid recirculating the pump (20) the pressure in the tank (4) is -0.1 barg and the outlet temperature of the stream (24) is 97 °C.

Claims

1. Procedure for continuous thermal hydrolysis of any type of organic matter that, to fully recover the energy and optimize the hydrolysis kinetics, comprises at least of stages: 1) feeding, stepped pressurization and sequential injection of low, medium and high pressure levels steam; 2) first stage of hydrolysis by consecutive steam explosion operations with the production of medium pressure level steam and thermal reaction; 3) second stage of hydrolysis consisting of steam explosion and low pressure steam production.

2. Process according to claim 1 and different from currently existing processes, is **characterized by** a step of feeding, pressurization and sequential injection of heating steam, **characterized by**:

   - use a stepped pressure increase system, which allows to receive and take advantage of different pressure levels steam. In any circumstance the steam coming from the tanks (3) and (4) are injected into the system. According to the laws of thermodynamics, this procedure allows a total recovery of energy, without the need to operate with high concentrations of organic matter.
   - use systems with at least two pumps, which when stepped pressurized, allow the proper injection of steam in the corresponding liquid-gas mixers.
   - due to the effect of staggering pressures, use centrifugal pumps instead of positive displacement pumps, which are more expensive and difficult to maintain.
   - reach temperatures and pressures on the feed to the hydrolysis stage (11) of up to 220 °C and 22 barg, higher than conventional.
   - use low pressure steam mixing systems that allow to lower the pressure on the chamber of the second flash (4) to values between 0 and -0.5 barg, with the consequent decrease in the outlet temperature of the hydrolyzed organic matter and its impact on net energy consumption.
   - use ultra-rapid systems for mixing live steam, so that the mixture to be hydrolyzed is only at a temperature higher than the secondary reactions' appearance temperature for less than 5 seconds. In such short times the extent of secondary reactions is negligible.

3. Process according to claim 1 and in a different way to those currently existing consists of a first hydrolysis step, **characterized by**:

   - use a sequence of steam explosion + thermal reaction stages, which improves the overall kinetics of the process by reducing the size of the facilities.
   - use a tank (3) that simultaneously operates as a flash tank, facilitating a first breakdown on the physical structure of organic matter, and as a reactor that facilitates the hydrolysis reaction by temperature.
   - apply the temperature hydrolysis reaction to sludge that has previously undergone a steam explosion process, with which the reaction kinetics is significantly increased, allowing operating at temperatures between 140 and 180 °C and reaction times of less than 15 minutes.

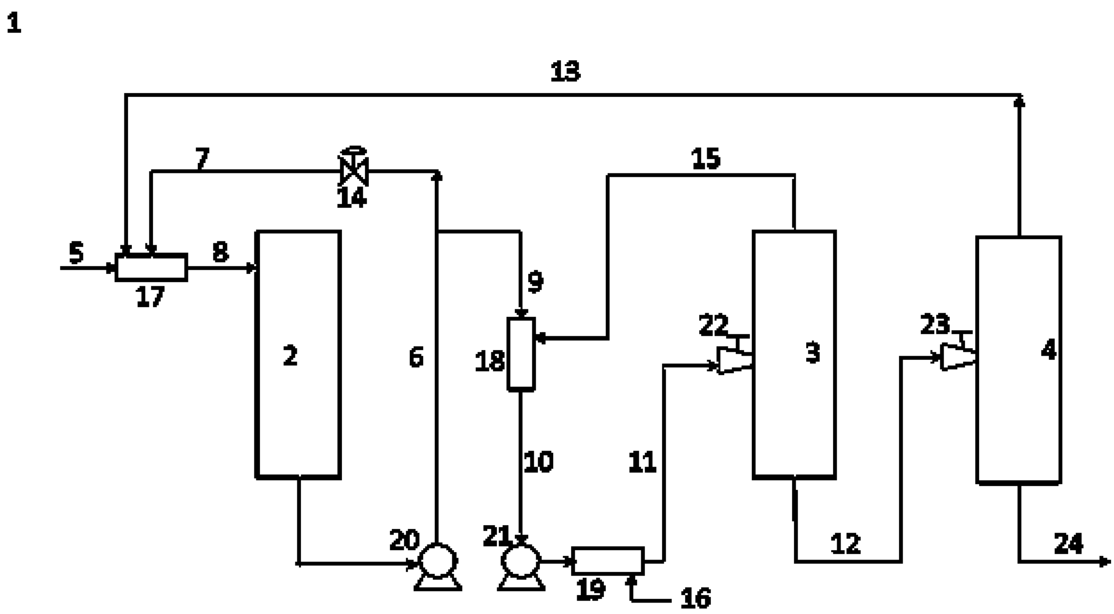
4. Process according to claim 1 and different to those currently existing, consists of a second hydrolysis step, **characterized by**:

   - making a second steam explosion from pressures between 3 and 10 barg up to pressures between -0.5 and 0.5 barg.
   - producing a low-pressure steam that is conducted to the stage 1 of claim 1.

5. A process that according to the preceding claims and in a different way to those currently existing operates not only continuously but also in steady state with the consequent stability of the process and ease of control, which gives the procedure a great operational robustness.

6. An installation (1) for continuous thermal hydrolysis of organic matter, which according to claims 1, 2, 3, 4 and 5 can treat any type of solids and is especially suitable for treating sludge produced in residual water treatment plants and that achieves energy self-sufficiency by operating with lower concentrations of dry matter than those required by existing technologies. The installation includes:

- at least two pumps (20) and (21) for the stepped pressurization of organic matter fed. In the first phase, the pressure of the stream (9) rises to values between 3 and 8 barg. In the second phase, the pressure of the stream (11) is raised to values between 10 and 22 barg.
- at least three static or dynamic fluid-steam mixers (17), (18), (19), which allow a stepped increase in temperature, taking advantage of at least the low (13) and medium (15) pressure steam produced in the flash stages.
- valve (14) that can act as a pressure or cut control valve.
- Mixer (17), which depending on the characteristics of the feed (5) can be an ejector, thereby reducing the tank pressure (4) to values between 0 and -0.5 barg.
- a mixer (19) that receives live steam and which, in order to prevent the occurrence of secondary reactions, operates with mixing times of less than 5 seconds.
- a stream (11) whose pressure values (up to 22 barg) and temperature (220 °C), far exceed the limits imposed by other technologies.
- decompression elements that can be selected between constrictions, nozzles or valves (22) and (23) that are dimensioned according to the flow rate to be treated, to produce the pressure drop generated by the steam explosion mechanism. In the device (22) the inlet pressures may vary between 10 and 22 barg, with a maximum outlet pressure of 8 barg. For the device (23) a maximum inlet pressure of 8 barg, the outlet pressure moves in a range between -0.5 to 0.5 barg.
- tanks (3) and (4) that operate as flash chambers in which medium pressure (15) and low pressure (13) steam are produced, which when condensed are used to increase the temperature of the organic matter to be hydrolyzed. Likewise, the tank (3) acts as a reactor. The operating temperatures in the tank (3) can be set between 140 and 180 °C, while in the tank (4) they are set between 80 and 110 °C.
- instrumentation and control systems, not indicated in figure 1, so that according to claim 5, which implies steady state, allow to pre-set and maintain constant the desired value of any of the operation variables at all points of the installation.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INTERNATIONAL SEARCH REPORT

International application No.
PCT/ES2017/070795

A. CLASSIFICATION OF SUBJECT MATTER

C02F11/18 (2006.01)
C02F1/02 (2006.01)
According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
C02F

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

EPODOC, INVENES, ESPACENET, INTERNET, NPL, WPIAP, WPI

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	WO 2015189449 A1 (TE CONSULTING HOUSE 4 PLUS SL) 17/12/2015, claims and figures	1-6
A	WO 2015032552 A1 (VEOLIA WATER SOLUTIONS & TECH) 12/03/2015, claims and figures	1-6
A	WO 2016079361 A1 (AQUATEC PROYECTOS PARA EL SECTOR DEL AGUA S A U) 26/05/2016, claims and figures	1-6

☐ Further documents are listed in the continuation of Box C.　　☒ See patent family annex.

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"&" document member of the same patent family

Date of the actual completion of the international search	Date of mailing of the international search report
19/03/2018	**(21/03/2018)**
Name and mailing address of the ISA/ OFICINA ESPAÑOLA DE PATENTES Y MARCAS Paseo de la Castellana, 75 - 28071 Madrid (España) Facsimile No.: 91 349 53 04	Authorized officer I. Abad Gurumeta Telephone No. 91 3495337

Form PCT/ISA/210 (second sheet) (January 2015)

INTERNATIONAL SEARCH REPORT

Information on patent family members

International application No.
PCT/ES2017/070795

Patent document cited in the search report	Publication date	Patent family member(s)	Publication date
WO2016079361 A1	26.05.2016	CN107074602 A	18.08.2017
		ES2570812 A1	20.05.2016
		ES2570812 B1	05.09.2017
		EP3222591 A1	27.09.2017
WO2015189449 A1	17.12.2015	MX2016016279 A	05.07.2017
		JP2017525557 A	07.09.2017
		US2017203988 A1	20.07.2017
		US9751791 B2	05.09.2017
		KR20170018012 A	15.02.2017
		EP3156374 A1	19.04.2017
		EP3156374 A4	14.06.2017
		ES2538176 A1	17.06.2015
		ES2538176 B1	05.10.2015
WO2015032552 A1	12.03.2015	RU2016112950 A	11.10.2017
		JP2016529103 A	23.09.2016
		CN105683098 A	15.06.2016
		US2016214880 A1	28.07.2016
		KR20160050027 A	10.05.2016
		AU2014317407 A1	21.04.2016
		CA2922777 A1	12.03.2015
		EP3041796 A1	13.07.2016
		FR3010403 A1	13.03.2015
		FR3010403 B1	13.01.2017

Form PCT/ISA/210 (patent family annex) (January 2015)

REFERENCES CITED IN THE DESCRIPTION

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Patent documents cited in the description

- ES 2570812 A1 **[0003]**
- ES 2538176 B1 **[0004] [0005]**