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INTENSIFICATION OF MAN-MADE WASTE METHANE FERMENTATION PROCESS IN COMPLEX FERTILIZER TECHNOLOGY

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Abstract. The methods of laboratory studies found the opportunity to intensify the process of methane fermentation which is used in the complex fertilizer technology. To intensify fermentation it was suggested to reprocess the mixture by chemical and mechanical ways that consists in the preliminary dispersion. Thus, the duration of mixture methanation process in mesophilic regime has been reduced by more than a half. The functions have been obtained and can be used to select the method of fermented mixture preprocessing in industry.

Keywords: complex fertilizer, man-made waste, dispersion, methanation.

1. Introduction

Due to the overall global economic crisis international prices for fertilizers have risen and make up the approximate cost, USD/t: nitrate – 380, phosphorus – 650, potassium – 450 [1-3]. The use of local anthropogenic waste [4, 5], containing nitrogen, phosphorus, potassium, calcium, in the technology of complex fertilizer manufacturing will lead to the increased productivity of agricultural plants and reduce the cost of expensive raw materials purchasing.

In several research works [3, 6, 7] the authors demonstrate the possibility and prospects of getting complex fertilizer by selecting different combinations of man-made waste and organic raw materials (brown coal, lignin, peat, *etc.*), the use of chemical and technological methods.

Nowadays complex peat fertilizers from different trade names of humate-containing products are produced non-granulated, with a low nutrients index (3–4 %). The percentage of moisture of this fertilizer type is high – up to 50 %. This fertilizer is produced sporadically in small batches [8].

The authors have developed the ways how to get and use [9] the organo-mineral fertilizer which is based on the thermal power plant (TPP) water demineralization sludge, which includes the sludge feed to the automatic

recessed plate filter-presses. But this fertilizer may be used only in acidic soils.

The existing technologies of complex fertilizer obtaining have drawbacks, particularly the duration of the process in a mesophilic methane fermentation mode exceeds 50 days [10]. It takes some time to reach the balance between the processes of oxidation and reduction. Also, the rate of methane fermentation significantly increases with a temperature rise. The optimum fermentation temperature is considered to be 323–328 K. The low speed of methane fermentation process started to inhibit proliferation of its use in practice of complex fertilizer obtaining.

Researchers [11] have developed an energy-saving technology to recycle agricultural waste production using the process of the thermophilic methane fermentation mode. But the processes with higher temperatures demand greater costs for electricity and gas that prevents their wide application.

The bioreactor with the mesophilic methane fermentation regime found the widest use in the technology of complex fertilizer obtaining. The mesophilic regime is less energy intensive, but it does not allow using smaller methane-tanks. When using the mesophilic regime there is a low rate of organic matter decay, the duration of fermentation is up to 50 days, thereby the required volume of structures increases [12]. The previous mechanical and chemical mixture processing which is subjected to methane fermentation will allow fertilizer obtaining for less time.

An active metabolic process and a high speed biochemical metabolism are achieved by continuous replenishment of maximum possible size of boundary surfaces between the solid and liquid phases [10]. It is predicted that the use of the dispersion processes followed by settling in order to increase the amount of dry residue in a mixture which is subjected to methane fermentation will speed up the fermentation process.

2. Experimental

A mixture of man-made products (calcium sludge, activated sludge, chicken manure) was decontaminated and stabilized by a mesophilic methane fermentation mode. According to the results of sanitary-chemical and toxicological studies of man-made waste, water demineralization sludge has the 4th rate of toxic wastes hazard. The TPP water demineralization sludge contains the following components relative to a dry matter, %: CaCO_3 – 75.00; CaSO_4 – 6.00; MgO – 4.80; Fe_2O_3 – 0.20; Al_2O_3 – 0.80; Mn – 0.00731; Cu – 0.00021; Zn – 0.00038; Ni – 0.00060 [13]. The quality of man-made waste which was used for the researches is presented in Table 1.

We investigated the effect of preprocessing for man-made waste mixture with calcium sludge and the following dispersion according to the intensity of methane fermentation processes, particularly the kinetics of biogas selection from the fermented mixture. The dispersion was done with the help of a rotatory milling disk disperser which has 32 notches, rotating with a frequency of 17 s^{-1} on a vertical shaft. The flow of fluid in the apparatus goes in the tangential direction by the friction of fluid against the disc, and the disc also creates an axial flow. The peripheral speed is 3.04 m/s, that due to a small disk size meets a high number of liquid oscillations (533 s^{-1}).

The laboratory plant for obtaining the complex NPKCa-fertilizer which is based on man-made waste consists of a measuring container (1), siphon pipe (2),

disperser (3), bioreactor (4), thermostatically controlled heater (5), plug hatches (6, 10), an airtight heat sealing cap (7), magnetic stirrer (8), polyvinyl chloride pipes (9, 12), glasses for biogas collection (11) and measuring cylinder (13) (Fig. 1).

After filling the measuring container (1) with man-made waste (activated sludge, TPP water demineralization sludge, poultry manure), the generated mixture was pumped out with a help of the siphon pipe (2) into the disperser (3). The disperser was on for 1 min. The homogenized solution of man-made waste was poured into the glass bioreactor (4) for fermentation. The glass bioreactor (4) of 1 dm capacity was sealed with a rubber plug (6), after that the pipe (9) for gas discharge, the glass for biogas collection (11) and the measuring cylinder (13) for measuring the capacity of water extruded by biogas were attached. The container for fermentation (4) was set up onto the magnetic stirrer (8) and covered with an airtight heat sealing cap (7). To maintain the constant temperature in a mesophilic fermentation regime a thermostatically controlled heater was used, and it was immersed into the bioreactor. Twice a day the mixer was turned on, the mixing intensity was 2.3 s^{-1} . There four methane fermentation plants were set up simultaneously.

For fermentation there was used a mixture of man-made waste with the following ratio relative to a dry matter, %: chicken manure – 76, activated sludge – 20, TPP water demineralization sludge – 4 [14]. A mixture of man-made waste was divided equally into four parts and loaded alternately into the bioreactors.

Table 1

The quality of man-made waste to produce complex NPKCa-fertilizer

Item	Activated sludge	Water demineralization sludge	Poultry manure
Ash content, %	13.76	56.19	21.03
Carbon content, %	30.00	9.00	30.00
Nutrient content, relative to a dry matter, %:			
N	9.80	30.00	3.56
P	2.10	0.50	1.70
K	0.15	0.03	5.10
Ca	0.001	24.17	5.33
pH, in conventional units	5.30	9.10	6.50
Moisture, %	92.00	58.43	55.17

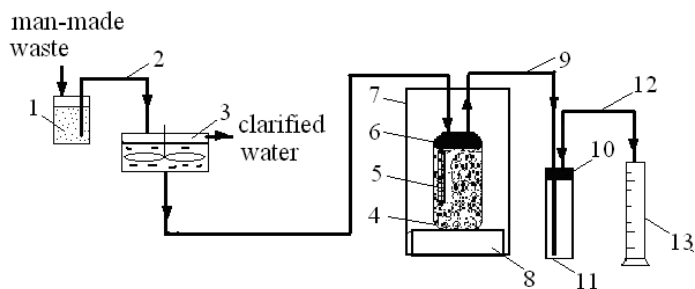


Fig. 1. The diagram of the laboratory plant for obtaining complex NPKCa-fertilizer which is based on man-made waste: measuring container (1); siphon pipe (2); disperser (3); bioreactor (4); thermostatically controlled heater (5); plug hatches (6, 10); airtight heat sealing cap (7); magnetic stirrer (8); polyvinyl chloride pipes (9, 12); glass for biogas collection (11) and measuring cylinder (13)

The first part was not processed additionally and was immediately loaded into the first bioreactor. The second part was processed with the calcium water demineralization sludge. After setting it out for 1.5 h, the residual matter was formed, and it was loaded into the second bioreactor. The third and the fourth parts of biomineral mixture were also processed with the calcium water demineralization sludge dosing of 40 mg/dm^3 and were dispersed for 1 min. The dispersed mixture was settling out for 2.5 h and then the formed compacted sediment was loaded into the third and fourth bioreactors.

The investigated mixture was loaded into the bioreactor; it was sealed with a plug which contained a pipe connecting the cylinder and gas receivers. The gas receivers were filled with water. The heater with a heating controller was installed into the cylinder. The cylinder was placed on a magnetic stir bar and covered with an airtight heat sealing cap. Then the cylinder and all the connections, channels were checked for leaks. Upon reaching the desired temperature in the bioreactor, the volume of the discharged gas in the cylinder was measured. The main distinguishing feature of the fourth bioreactor was the fact it was not heated additionally in contrast to the previous three ones. The biogas volume was measured by the volume of water displaced from the receiver to the gas cylinder. The daily volume of formed biogas in the graduated cylinder with water was controlled every day. The experiment lasted for 30 days.

3. Results and Discussion

During the first 12 days biogas discharge in all four bioreactors was increasing with different pace (Fig. 2). In contrast to the research which was carried out by the authors [15], the study of biogas obtaining from organic-mineral mixtures at high temperatures without previous chemical and mechanical processing of the fermented mixture, the biogas selection kinetics in the researched plants is much higher.

This experiment indicates that after the 20-th day of the research the biogas discharge in all four bioreactors got reduced essentially and it becomes impractical to obtain it. However, the total amount of selected gas during 20 days is higher than the total amount of gas received by the authors [15] during 50 days.

It was found out that the use of the preliminary dispersion of the fermented mixture reduces fermentation time from 50 to 20 days. The fermentation process at four plants had different speeds of biogas discharge. At all plants the maximal biogas discharge (from 1 kg of dry matter) was observed on the eleventh or thirteenth days: 0.034 m^3 – at the plant without preprocessing of mixture (Fig. 2, *curve 1*); 0.147 m^3 – with the mixture processed with calcium sludge (Fig. 2, *curve 3*); 0.165 m^3 – with the mixture processed

with calcium sludge and dispersed (Fig. 2, *curve 4*); 0.1 m^3 – with the mixture processed with calcium sludge and dispersed without further heating (Fig. 2, *curve 2*). After the twelfth day of the experiment, the biogas discharge at all four plants is getting variably smaller: less than 0.005 m^3 – at the plant without the mixture preprocessing (Fig. 2, *curve 1*); less than 0.015 m^3 – with the mixture processed with calcium sludge (Fig. 2, *curve 3*); less than 0.04 m^3 – with the mixture processed with calcium sludge and dispersed (Fig. 2, *curve 4*); less than 0.024 m^3 – with the mixture processed with calcium sludge and dispersed without further heating (Fig. 2, *curve 2*).

It is known that the activity of methane generating bacteria increases under alkaline conditions. To maintain the metabolic activity of methane bacteria on a constant basis it is necessary to maintain pH not lower than 6.5. In the previous chemical processing of the fermented mixture with calcium TPP water demineralization sludge the pH level in the initial substrate increases. The use of TPP water demineralization sludge simultaneously leads to $\text{pH} \leq 8.5$ aligning and intensifies the process of methane fermentation.

The mathematical description of the process to obtain complex fertilizer from raw materials of man-made waste was based on the fact that during the process of methane fermentation the anaerobic microorganisms increase, for which the organic substance with a simultaneous organic rate is a staple food. Since there are two mutually exclusive processes, the maximum speed of fermentation process with higher volume of biogas discharge is observed. In Fig. 3 the accumulation of outgassing kinetics from the man-made waste mixture according to time is observed: *curve 1* – non-processed additionally mixture; *curve 2* – preprocessed with calcium sludge and dispersed without further heating; *curve 3* – processed with calcium sludge; *curve 4* – processed with calcium sludge and dispersed.

For the obtained models the coefficient approximation value (R^2) (Fig. 3) is close to 1, indicating the high approximate quality of the constructed models. The adequacy test of models was carried out for accuracy.

In order to develop recommendations for four plants it is advisable to conduct a comparative analysis of the research results. For this purpose, mathematical modeling of man-made waste mixture methane fermentation was conducted by regression analysis method which is based on the data received from the laboratory tests.

As a result there are equations which describe the process of the kinetics to accumulate discharged biogas for four plants:

a) additionally unprocessed

$$\hat{y} = 0.021x - 0.0365 \quad (1)$$

b) processed with calcium sludge

$$\hat{y} = 0.0951x - 0.133 \quad (2)$$

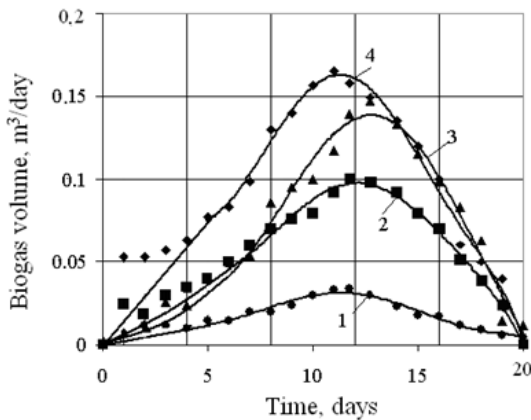


Fig. 2. Biogas selection kinetics from fermented mixture vs. time relative to 1 kg of dry matter: unprocessed (1); processed with calcium sludge and dispersed without further heating (2); processed with calcium sludge (3) and preprocessed with mixture of calcium sludge and then dispersed (4)

c) processed with calcium sludge and dispersed

$$\hat{y} = 0.1149x - 0.1498 \quad (3)$$

d) preprocessed with calcium sludge and dispersed without further heating

$$\hat{y} = 0.0694x - 0.1103 \quad (4)$$

The adequacy verification for the model that describes the known method of fermentation (Fig. 3, curve 1) is performed by using Fisher's criterion. The estimated values of the criterion were obtained by the formula [16]:

$$F_p = F(1, n-2) = \frac{(n-2) \cdot \sum_{i=1}^n (\hat{y}_i - \bar{y})^2}{\sum_{i=1}^n (y_i - \hat{y}_i)^2} = 767.334; \quad (5)$$

$$n = 20$$

$$\hat{y} = 0.021x - 0.0365$$

$$\bar{y} = \frac{\sum_{i=1}^n y_i}{n} \quad (6)$$

where y_i – data of laboratory researches.

The critical criterion value at 0.95 level of the value was received by Fisher's allocation tables, using the opportunities of Microsoft Excel spreadsheets:

$$F_{crit} = F(a, K_1, K_2) = 4.14 \quad (7)$$

$$a = 0.05; K_1 = 1; K_2 = n - 2 = 20 - 2 = 18$$

As $F_p > F_{crit}$, so, it may be claimed with a probability of 0.95 that the mathematical model is adequate to the experimental data. For similar verification of the mathematical model of biogas discharge kinetics of the fermented mixture processed with calcium sludge

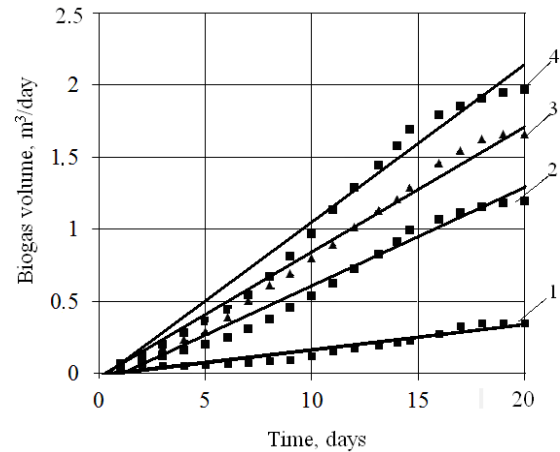


Fig. 3. The accumulation of outgassing kinetics from man-made waste mixture vs. time relative to 1 kg of dry matter: unprocessed additionally (1); preprocessed with calcium sludge and dispersed without further heating (2); processed with calcium sludge (3) and processed with calcium sludge and dispersed (4)

$F_p = 2911.16$, the correlation $F_p > F_{crit}$ is performed again, indicating that the biogas discharge kinetics model (Fig. 3, curve 3) $\hat{y} = 0.0951x - 0.133$ of man-made waste mixture which was additionally processed with calcium sludge is adequate to the experimental data.

To verify the adequacy of model $\hat{y} = 0.1149x - 0.1498$, that describes the biogas discharge kinetics of the fermented mixture which was processed with calcium sludge and dispersed (Fig. 3, curve 4) we calculate $F_p = 969.12$. The performance of the correlation $F_p > F_{crit}$ confirms the adequacy of the model.

The similar calculations were done to verify the model $\hat{y} = 0.0694x - 0.1103$, which describes the dynamics of the biogas discharge of the fermented mixture which was processed with calcium sludge without further heating (Fig. 3, curve 2), the calculated criterion value $F_p = 1008.94$, that indicates the adequacy of this model to the research data.

The combination of preprocessing with calcium sludge and dispersion allows increasing the biogas discharge kinetics of the fermented mixture and increases the total volume of the discharged biogas from 0.35 to 2.01 m³ relative to 1 kg of dry matter (Fig. 3). The first factor in the equations (Fig. 3) determines the speed of gas accumulation according to time. The analysis of constructed models suggests that biogas obtaining from the mixture which was pre-processed with calcium sludge and dispersed with further heating is the most optimal (Fig. 3). The technology of biogas obtaining from the processed calcium sludge mixture which was non-dispersed previously and heated is almost equal. At the same time, the known technology of biogas obtaining

from a mixture which was not processed with calcium sludge and was not dispersed with further heating is the least productive. Compared with the known technology of biogas obtaining from the mixture preprocessed with calcium sludge and dispersed without heating, this one is much better. Therefore, for electricity saving it is advisable to use such a technology of biogas obtaining in the warm season.

The complex NPKCa-fertilizer which was obtained by adding TPP water demineralization sludge, by man-made waste dispersion and by heating the fermented mixture in the mesophilic mode has a high level of quality: the moisture content – 40 %; the ash-content – 34 %; pH – 8,5; the nutrient content, %, relative to dry matter: N – 4.54; P – 3.55; K – 4.11; Ca – 14.62; C – 26.65. The micro-element content in the obtained fertilizer, mg/kg, relative to a dry matter: Mn- 70.69, Cu – 20.97, Zn – 410.0, Fe - 2 547.5, Ni – 12.47, Co – 1.93, Pb – 15.34, Cd – 0.34, Hg – 4.8, Cr – 5.7 [17]. Such micro-element content does not exceed the maximum allowable concentration in the soil [18].

Thus, Fig. 4 shows the change of the general texture of man-made waste before and after dispersion, followed by the methane fermentation. After dispersing and methane fermentation the man-made waste mixture (Fig. 4c) had smaller fraction and became more free-running.

The biogas which was obtained from all four plants has the density of approximately $1.2 \text{ m}^3/\text{kg}$, up to 60 % of

methane and about 40 % of carbon dioxide, excluding other impurities, such as hydrogen sulfide, nitrogen, oxygen and others [17]. The content of which does not exceed 1 %, it burns well with blue flame, and with the corresponding odor. This allows using it in the transport industry, or municipal engineering.

Relying on the results of the research there was created a general technology for complex NPKCa-fertilizer based on man-made waste, which can be implemented on the basis of the classical treatment facilities where a stage of biological wastewater filtering exists. Fig. 5 shows the fundamental technological scheme how to obtain the complex NPKCa-fertilizer based on man-made waste.

The fundamental technological scheme how to obtain the complex NPKCa-fertilizer based on man-made waste includes a disperser (1), where activated sludge and poultry manure are dispersed.

Reynolds number for disperser (1) has to be $17.47 \cdot 10^3$, the dispersion duration under these conditions should be in the range from 0.5 to 1.5 min.

The mixture of the dispersed activated sludge and poultry manure enters the dirt collector (2). To speed up the process of settling, the TPP water demineralization calcium sludge is added dosing $40 \text{ mg}/\text{dm}^3$. In the dirt collector (2) there dispersion stratification of solid phase in water can be seen. The process of settling should take from 2 to 2.5 h.

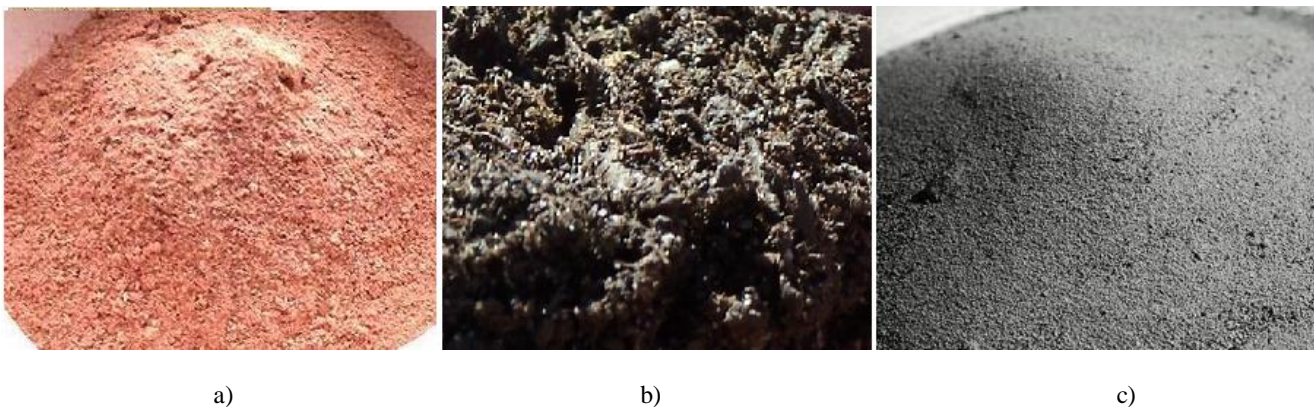
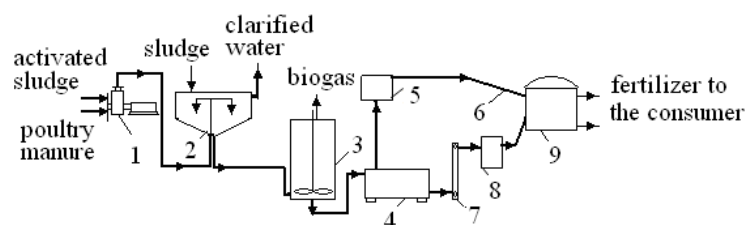


Fig. 4. The general view of man-made waste before and after processing with dispersers and methane fermentation: mixture of TPP water demineralization calcium sludge without processing (a); poultry manure without processing (b) and mixture of TPP calcium sludge, activated sludge, poultry manure after dispersion and methane fermentation (c)

Fig. 5. The fundamental technological scheme how to obtain the complex NPKCa-fertilizer based on man-made waste: disperser (1); dirt collector (2); bioreactor (3); centrifuge (4); section for dosing and packaging of liquid fertilizer (5); conveyor belt for liquid fertilizer (6); elevator (7); section for dosing and packaging of complex NPKCa-fertilizers (8) and warehouse of ready-for-use fertilizer (9)



The residue formed in the dirt collector (2), is pumped to the bioreactor (3) for joint methane fermentation of all the complex NPKCa-fertilizer components. It is recommended to use the mesophilic mode of methane fermentation and the process duration should be about 20 days. After the fermentation process the fermented mixture is fed to the centrifuge (4), where under the centrifuge rotates within 83–117 s⁻¹ during 260–280 s the complex NPKCa-fertilizer is dehydrated. The created concentrate according to its composition corresponds to a liquid fertilizer, so it is suggested to be dosed and packed into plastic drums on the section for dosing and packaging of liquid fertilizer (5). The packaged liquid fertilizer is transported by the conveyor belt for liquid fertilizer (6) to the warehouse of ready-to-use fertilizer (9). The dehydrated complex NPKCa-fertilizer is transported by the elevator (7) to the section for dosing and packaging of fertilizer (8), from which the fertilizer in plastic bags is unloaded at the warehouse of ready-to-use fertilizer (9).

4. Conclusions

On the basis of laboratory studies the possibility of methane fermentation process acceleration at chemical and mechanical preprocessing of man-made waste mixture, including calcium sludge processing and dispersion was proved. The less effective technology for complex fertilizer from man-made waste there is the method of mixture preprocessing with calcium sludge and dispersion without further heating, but compared to the known method without preprocessing, it may be used in the period from May to September, which will be energy saving.

The influence of preprocessing and calcium sludge dispersion of the fermented man-made waste mixture on the kinetics and the biogas discharge speed was discovered by the regression analysis method and laboratory experiments. With a probability of 0.95 (according to Fisher's criterion) it is possible to claim that the constructed mathematical models are adequate to the experimental data.

It was established that the previous dispersion of the man-made waste which is fermented, speeds up the methane fermentation process twice and increases the volume of discharged biogas from 0.35 to 2 m³ per 1 kg of dry matter. During the dispersion of man-made waste the boundary surface replenishment between the solid and liquid phases takes place, providing high speed of biochemical metabolism during the next methane fermentation. And the additional chemical processing of fermented raw materials with calcium sludge helps to maintain the pH rate at the level of 8.5 and permits to create the environment where methane-producing bacteria become active and to accelerate methane fermentation from 50 to 20 days.

It was reasoned and proved that there is a possibility to use man-made waste such as calcium sludge, and activated

sludge as the secondary raw material to obtain the new complex NPKCa-fertilizer of high quality relative to dry matter %: N – 4.54; P – 3.55; K – 4.11; Ca – 14.62; C – 26.65 containing the amount of micro-elements that does not exceed maximum allowed concentration.

The fundamental technological scheme how to obtain the complex NPKCa-fertilizer based on man-made waste was developed, in which the dispersion process is applied for the first time that accelerates the settling and the subsequent methane fermentation processes twice.

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ІНТЕНСИФІКАЦІЯ ПРОЦЕСУ МЕТАНОВОГО БРОДІННЯ ТЕХНОГЕННИХ ВІДХОДІВ В ТЕХНОЛОГІЇ КОМПЛЕКСНОГО ДОБРИВА

Анотація. Методами лабораторних досліджень встановлено можливість інтенсифікації процесу метанового бродіння, що застосовується в технології одержання комплексного добрива. Для інтенсифікації бродіння запропоновано проводити попереднє хімічне й механічне оброблення суміші, що полягає у попередньому диспергуванні. Це дало можливість більш ніж у двічі скоротити у мезофільному режимі тривалість процесу метанування суміші. Отримано математичні залежності, що можуть використовуватись для вибору способу попереднього оброблення зброджувальної суміші в промисловості.

Ключові слова: комплексні добрива, техногенні відходи, диспергування, метанування.