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Technology for conversion of whey into organic-mineral fertilizers with amino acids

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Rational processing of whey is one of the most important problems of the dairy industry. Less than 50% of this waste is processed in Russia. Unprocessed whey is discharged into the sewage systems, which significantly increases the degree of contamination of the process waste water. Basically, processing of whey in commodity products is limited by economic factors. At the enterprises of small and average capacity high costs of introducing processing technologies do not pay off proceeds from realization of the received products. The authors of the research have used the method of cryogenic concentration (freezing) which provided the complete conservation of bioactive substances (proteins, vitamins, enzymes) in whey. Proteins of the received concentrate were subjected to enzymatic hydrolysis to free amino acids. The mineral salts complex (which contains plant-available forms of nitrogen, phosphorus, potassium, magnesium and trace elements) was dissolved in the hydrolysate. As a result, the researchers have got a marketable product – liquid organic-mineral fertilizer with natural amino acids. Whey-fraction that remained after cryogenic concentration contained no more than 1.2% of dry substances. For its removal there was used glauconite-containing efel as sorbent (the waste of phosphorite ore enrichment, it is a quartz-glauconite sand containing up to 45% glauconite). The waste sorbent was dried up and used as the second commercial product – solid organic-mineral fertilizer (S-OMF). The S-OMF included only natural ingredients, which made this product suitable for use as a fertilizer in natural farming systems.

The simplicity and high market value of the received marketable products make it economically viable to introduce the proposed technology into practice. The calculated payback period of the offered technology at the enterprises of a mean power is less than two years.

Keywords: whey, organic-mineral fertilizers, fertilizers with amino acids, sorbents, glauconite, non-waste technologies.

Whey is the main residue left after the processing of milk into cheese (cheese whey), curd (curd whey), casein (casein whey). The chemical composition of the residue mainly depends on the quality of the source raw material, the technology of its processing, and the characteristics of the products obtained. The main components of whey are lactose, proteins, fats, organic acids, inorganic compound and vitamins. The total concentration of solids in whey is on average 5.5–7.0%, i.e. more than 40% of the dry substances contained in natural cow milk become residue. The valuable chemical composition makes whey a promising raw material for recycling. Currently, there are technologies that allow to process whey into dry or condensed foods, to isolate lactose, protein components, vitamins from lactose, to produce lactic acid, ethanol, food and feed additives, sports and health food supplements, various drinks, biodegradable polymers, nutrient media for microorganisms and other products [1–3]. Despite the wide possibilities for organizing whey processing, the problem of rational utilization of residue is still far from the solution and remains one of the most important problems of the dairy industry. The transition to non-waste technologies is a fundamental element of the successful development of the national economy and the most important condition for protecting the environment from pollution [4].

According to various estimates, Russia process from 20 to 50% of whey. Unprocessed whey goes to the sewage system, which leads to a significant increase in the degree of contamination of technological waste water of milk processing plants with biodegradable substances [5–7]. The discharge of whey into the sewage system causes a sharp increase in the chemical and biochemical consumption of runoff oxygen, since up to 50 dm^3 of oxygen is necessary to oxidize 1 dm3 of whey in sewage waters [8]. Pollution of sewage with whey leads to disruption of the normal operation of treatment facilities, as this residue worsen sedimentation properties of activated sludge [9, 10].

Volumes of unutilized whey can be estimated from the amount of cheese and cottage cheese produced in the country. In 2015, the Russian Federation produced 1375 thousand tons of cottage cheese, cheese and cheese products [11]. In 2016 and 2017, according to the Milknews Analytical Center, the volume of cheese production slightly increased (by 2% in 2016 and 4% in 2017) [12]. With such volumes of production, the mass of whey should be 11.5 million tons. Assuming that about half of whey (about 6 million tons) goes into the sewage system, the level of environmental pollution is very significant. Economic losses from the loss of valuable secondary raw materials is also very significant, as the loss of 6 million tons of whey is equivalent to a loss of 2.5 million tons of milk (8% of the total milk produced in Russia in 2016).

Analysis of the current situation shows that the implementation of technologies for the complete processing of whey has economic prospects only at large dairy processing enterprises. The introduction of appropriate technologies at small enterprises is limited by such factors as high costs for the acquisition and maintenance of equipment; extremely limited shelf life of whey; low content of solids; complex and unstable chemical composition of the corresponding secondary raw materials; a limited sales market for whey processing products; insignificant profit in comparison with expenses for residue processing. Inclusion of natural whey in rations of agricultural animals is also usually not justified due to the high costs of transporting a low-concentrated product and its rapid microbiological damage.

This situation requires the development of new technologies of whey recycling suitable for small and medium-sized enterprises. One of the possible directions of processing whey can be its use for the production of organomineral fertilizers. A number of domestic and foreign scientists prove the prospects of this direction in their researches [13–16].

Features of the chemical composition make it possible to use whey as a source of amino acids and other biologically active substances that have a positive effect on the growth and development of plants (succinic and chlorogenic acids, vitamins, enzymes, carbohydrates, etc.). The demand for fertilizers with biologically active components has increased significantly in recent years. The use of such fertilizers contributes to an increase in the yield of agricultural plants, ensure high quality of cultivated products, shorten maturation periods, increase plant resistance to unfavorable environmental conditions [17, 18]. Prices for fertilizers containing amino acids as biologically active components are significantly higher than prices for such products as whey powder or food lactose. In 2017 wholesale prices for food lactose were 60–80 rubles/kg, for whey powder – 45–60 rubles/kg. At the same time fertilizer with Aminozol amino acids (Lebozol Dünger GmbH, Germany) was sold at a price of 990 rubles for 500 ml; fertilizer Quantum-AminoMax (Amino acids) (NPK "Kvadrat", Ukraine) at a price of 550 rubles per liter; Radifarm (Valagro, Italy) at a price of 462 rubles for 100 ml [12]. Thus, the development and implementation of low-cost technologies for the production of organo-mineral fertilizers with amino acids based on whey can be of great interest to enterprises both from the ecological and economic point of view.

The purpose of the research is to develop a technology for processing whey into organo-mineral fertilizers with amino acids suitable for use in low and medium milk processing plants.

The tasks of the research are:

– to develop a non-waste technological scheme for processing whey into organo-mineral fertilizers containing a complex of amino acids and other biologically active substances. To ensure the maximum extraction of solids from whey, the process flow diagram should include a step of concentrating the initial waste in order to process the concentrate obtained into a liquid organomineral fertilizer (L-OMF) and a sorptive post-treatment of a low concentration whey fraction, followed by using the spent sorbent as a solid organomineral fertilizer (S-OMF).

– to determine the optimal way of preparing the components extracted from whey in order to add liquid organomineral fertilizers in the composition ;

– to determine the composition of mineral components that allow to obtain a balanced and stable organomineral fertilizer during storage;

– to choose a sorbent for post-treatment of low-concentration whey fraction. The spent sorbent should be suitable for use as a fertilizer;

– to perform calculations that allow to assess the economic feasibility of introducing the developed technology into practice.

Materials and methods

For our experiments we used curd whey obtained from one of the milk processing enterprises of the Kirov region. Table 1 gives data on the chemical composition of whey.

Table 1

Chemical composition of curds

Indicators	Value	Methods of analysis
Acidity active, pH	4.9±0.1	Potentiometric method, according to GOST 32892-2014. Milk and dairy products. Method for measuring active acidity (with amendment)
Acidity total (titrated), °T	69±2	Titrimetric method, according to GOST 3624-92. Milk and dairy products. Titrimetric methods for measuring acidity
Total solids content, %	6.8±0.4	Gravimetric method, according to GOST 3626-73. Milk and dairy products. Methods for measuring moisture and dry matter
Protein content, %	1.7±0.1	Kjeldahl method, according to GOST R 53951-2010. Dairy products, dairy components and milk-containing products. Determination of the mass fraction of protein by the Kjeldahl method
Sugar content, %	4.8±0.2	Iodometric method, according to GOST R 54667-2011. Milk and milk processing products. Methods for determining the mass fraction of sugar
Fat content, %	0.12±0.01	Acid method, according to GOST 5867-90. Milk and dairy products. Methods for measuring fat
Calcium content, мг/кг	480±14	Titrimetric method, according to GOST R 55331-2012. Milk and dairy products. Titrimetric method for measuring calcium content

Table 2

Bacteriological characteristics of whey sample*

Microorganisms	Duration of storage, days	
	2	4
	Number of microorganisms, thousand cells / cm^3	
Lactic Acid Bacteria		
– mesophilic	4984	9915
– aroma forming	1.13	0.98
– thermophilic	45.92	58.8
Acetic acid bacteria	0.62	1.47
Group of *E. coli*	4.81	6.47
Yeast, mold micromycetes	894	4497

Note: “” – microbiological research of whey was made according to standard methods used in the dairy industry [19].*

Table 2 gives data on the microbiological contamination of whey used in the research.

Microbiological composition of whey is quite diverse. It leads to rapid deterioration of residue during storage in processing conditions. However, microorganisms found in whey are not dangerous in terms of bacterial pollution of the environment. It should be noted that the composition of whey microflora depends on the spectrum of microorganisms present in the source raw material, the leaven used, features of the technological process and compliance with the sanitary standards for the production of dairy products [20].

To increase the content of dry substances in whey, the cryoconcentration (freezing) method was used. The initial whey was frozen at -15 °C. When 85–87% of the whey get solid, the remaining (unfrozen) part was drained and used for further processing into a liquid organomineral fertilizer. The content of dry matter in the unfrozen fraction (concentrate) was 30±2%. We chose the method of cryoconcentration due to the fact that, unlike evaporation and drying methods, low-temperature technologies allow to provide the most complete preservation of biologically active substances (proteins, vitamins, enzymes) contained in whey [21].

While melting, the frozen part of whey was divided into two fractions with a solids content of 6.0 to 0.4% and 1.2 to 0.1%. A more concentrated fraction was used for further concentration by the freezing method [22]. A less concentrated fraction was passed through a sorption column to maximize the removal of solids. The fraction of the low concentration fraction was 10% of the total mass of the cryoconcentrated whey.

As a sorbent for removal of residual solids from whey, we used the glauconite-containing efel – a waste of phosphate after processing at the Verkhnekamsk phosphorite mine. The composition of the efel included (% by weight): Al_2O_3 – 2.3; MgO: – 0.9; K_2O – 1.4; Fe_2O_3 – 4.1; SiO_2 – 49.3; CaO – 41. The content of glauconite in the ephel was 40±5%. The content of heavy metals (Cu, Zn, Hg, Cd) and arsenic did not exceed the content of these elements in phosphorite flour. To obtain data on the elemental composition of the efel, we used an atomic-emission method with inductively coupled plasma.

The choice of a sorbent for isolating dry substances from a low concentration whey fraction was due to the following reasons:

– composition which allows to use efel as agro-ore;

– size of particles allows to use it as a sorbent;

– low cost and availability;

– sorption properties concerning a wide range of organic and inorganic components;

– having calcareous components in the sorbent composition which are necessary for neutralization of acids contained in whey.

During the sorption of solids from whey, the glauconite-containing sorbent was enriched with nitrogen-containing components, which increased the value of this material as a fertilizer.

The spent sorbent was dried in a thermostat at a temperature of 104±1 °C to a residual moisture content of 11–12%. The dried form was a loose material suitable for adding to the soil without further processing.

For the hydrolysis of proteins in whey concentrate, a chymotrypsin enzyme was used [23]. Before adding the enzyme, the concentrate was neutralized with a solution of potassium hydroxide to pH 7.5. The choice of chymotrypsin is due to the high selectivity of this enzyme to whey proteins, as well as the virtual absence of analogues with necessary properties and affordable cost.

Potassium sulfate (source of potassium and sulfur), potassium dihydrogenphosphate (source of potassium and phosphorus), ammonium nitrate (source of nitrogen), magnesium sulfate (magnesium source), were used as the mineral components included in the hydrolyzate of whey; and sulfates of copper(II), zinc and manganese (II) were sources of microelements. The amino acids obtained during the proteolysis of whey proteins allowed the transfer trace elements to a complex-bound (chelate) state, which increased their biological activity [24].

Mineral components were added to the whey hydrolyzate in a crystalline form and mixed until the crystals dissolved completely. The prepared composition was settled for 24 hours and then filtered with a vacuum filter.

Results and discussion

The finished form of liquid organomineral fertilizer obtained by the proposed method was a clear solution of turquoise color with a light smell of whey. The total content of dry substances was 56±3%. The pH of the finished form was within 5.0–5.5. The content of amino acids determined by the method of high-performance liquid chromatography was 4.1±0.3%.

The introduction of mineral salts into whey hydrolyzate provided the effect of conservation of the organic matrix of L-OMF. In the conditions of high osmotic pressure created by a complex of mineral salts, the reproduction of bacterial flora is impossible, therefore the final product was not subjected to microbiological damage. Fertilizer was stable during storage for 6 months at a temperature of 22±3 °C.

Based on the results of laboratory experiments, we created a technological scheme for processing whey into liquid and solid organomineral fertilizers (Fig.).

According to the proposed scheme, whey, which is formed during the processing of milk, is sent to a special container – a storage reservoir of a 24-hour whey stock with a volume of 50 m^3. From the storage tank, whey is pumped into a tank for freezing. Freezing is carried out by immersion coils at the temperature of direct brine -20 °C and reverse brine -17 °C. Freezing of the main body of water occurs within 8–10 hours, while the dissolved substances in whey are concentrated in the liquid phase. The resulting concentrate is drained into a pre-freezer with a volume of 30 m^3. The freezer is equipped with coils, which are fed with a cold brine to freeze the remaining water for 2–4 hours. The brine heated in the freezer is sent to a heat exchanger-evaporator of an ammonia refrigerating machine with a cooling capacity of 800 kW for cooling in the forced circulation circuit.

After freezing the water and draining the unfrozen concentrate, a hot brine is fed into the pre-freezing coil to thaw the ice. The cooled brine is sent to a heat exchanger in which it is heated by heating water to 50 °C. From the heat exchanger, the brine is fed into the forced circulation circuit of the hot brine. The thawed ice is sent to a melt water collector with a volume of 40 m^3.

Drain of the unfrozen whey concentrate into a collector occurs with the help of a heating coil. The volume of the collection is 10 m^3. The concentrate, heated to room temperature, by a centrifugal pump is pumped to the fermentation hydrolysis reactor with a volume of 10 m^3.

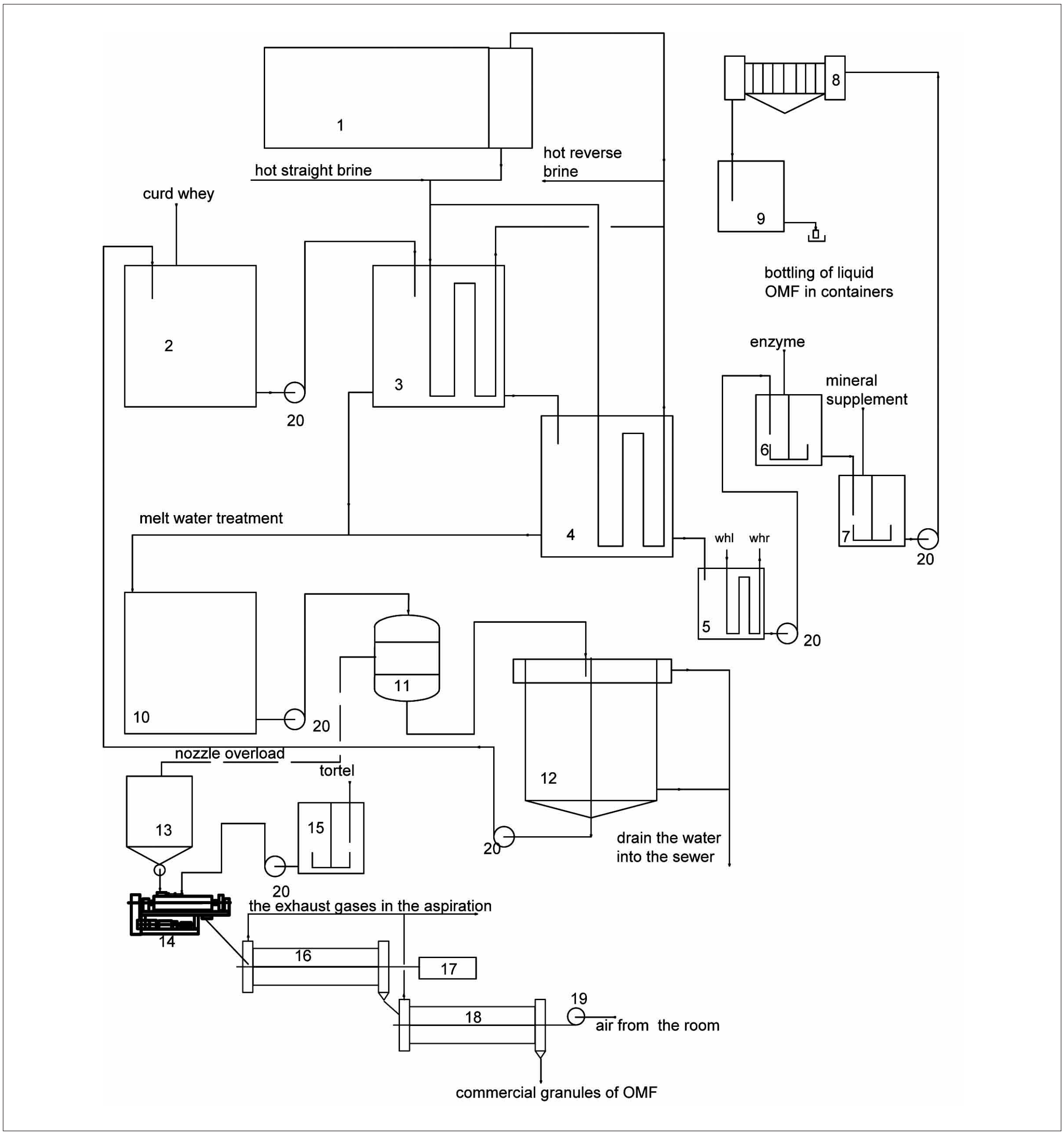

Fig. Technological scheme for processing of whey into organic-mineral fertilizers with amino acids: 1. Ammonia refrigerator. 2. Collector of the initial serum. 3. 1st stage freezer. 4. 2nd stage freezer. 5. Collector with concentrate preheater. 6. Reactor of enzymatic hydrolysis. 7. Reactor additives. 8. Filter press. 9. Collection of liquid OMF 10. Collector of thawed whey. 11. Packing filter with glauconite. 12. Radial settler. 13. Hopper waste bunker. 14. Turbofast mixer-granulator. 15. Collector of peat binder. 16. Drum dryer granules. 17. Gas heater. 18. Drum-freezer granules. 19. Blowing fan. 20. Pump.

The reactor is equipped with a stirrer, which allows to mix the concentrate with the addition of components neutralizing excess acidity and proteolytic enzymes. Dosage of alkaline components and enzyme preparations is carried out manually. The hydrolyzate from the reactor is drained into a collector with a mixer of 10 m^3 volume for mixing with mineral salts. Mineral salts in the hydrolyzate are dosed by hand. The components are mixed until the salts are completely dissolved.

The resulting liquid fertilizer is pumped by a centrifugal pump to a filter press for control filtration. The filtrate is fed to the pressure tank, from which it is bottled.

The melt water (low concentration whey fraction) is pumped out for sorption cleaning by a centrifugal pump. Removal of the residual quantity of the dissolved components from this fraction is carried out by the method of filtration under pressure on a packed filter with a glauconite ephel nozzle. The volume of the packed filter is 5 m^3, the diameter is 2 m.

Before draining into the sewage system, the filtrate is sent to a sump with a volume of 50 m^3. The resulting sediment from the cone bottom of the settler is pumped out to the collection of 24-hour storage of whey, and the clarified water is drained into the sewage system.

The spent sorbent from the packed filter is discharged into the collector and fed to a turbo-plastic mixer-granulator for producing fertilizer grits (S-OMF). As a binder at this stage, peat gels are used (peat processing product by the method of mechanical activation). The resulting grits are dried in a drum dryer and then cooled from 100 °C to 40 °C in an air cooler. The finished product is packaged in bags and sent for sale as a granular organomineral fertilizer.

The feasibility of implementing the proposed technology in production is confirmed by calculations. Assuming that the company will process 50 tons of whey per day and receive 10 tons of L-OMF, the payback period of all costs will be about two years, and the net profit from the sale of fertilizers is more than 20 million rubles a year. The corresponding calculations are given below.

Cost of the project are:

– costs for the acquisition and installation of equipment – 25.6 million rubles (the cost of a refrigeration unit is 10 million rubles; capacity and heat exchange equipment, circulating pumps is 6 million rubles; electrical equipment, instrumentation, automation, water supply and water disposal is 9.6 million rubles);

– costs of raw materials (excluding whey) are 100 thousand rubles per ton of finished product (mineral components, enzymes, sorbent);

– rent is not included in the cost of the project, as it is assumed that there are free production areas in the enterprise.

Income of the project is:

– reduction of payments for dumping of whey into the sewerage (according to calculations, the cost of daily dumping of fifty tons of whey into the sewer is 9.4 million rubles per year and includes water costs for dilution of whey up to normative indices of the pollutants content, as well as the cost of processing sewage effluent at sewage treatment plants of communal systems);

–revenue from the sale of L-OMF is approximately 912.5 million rubles per year (sale of 10 tons of L-OMF per day at a price of 250 000 rubles per ton during the year). If we assume that the net profit from the fertilizer sales does not exceed 10% (taking into account the costs of raw materials, energy carriers, staff salaries, tax deductions, product certification, etc.), and the volume of fertilizer sales does not exceed 10% (since the demand for this fertilizer is not fully understood and additional expenses for marketing activities will be required, as well as the development of a dealer network), then in this case the net profit of the enterprise will be about 10–12 million rubles a year.

The total income from the sale of fertilizers and the reduction of payments for the discharge of whey into the sewage system is approximately 20 million rubles per year, i.e. all the cost of equipment, documentation and marketing preparation of a new product should be paid off in about 18 months.

Additional income can be obtained from the sale of S-OMF. It can be used as a natural fertilizer for local needs.

Conclusion

The present research shows that whey is a valuable raw material for processing it into organomineral fertilizers containing a complex of biologically active substances (amino acids, vitamins, enzymes, salts of organic acids, etc.). To increase the concentration of dry substances in whey, the cryoconcentration method can be used which prevents the destruction of the corresponding organic components in the source material and increases the dry matter content to 30%. The enzyme chymotrypsin can be used for the hydrolysis of protein components in the cryoconcentrate.

Unlike other types of organic raw materials used for the production of organomineral fertilizers with amino acids, whey contains lactose. The presence of lactose in the whey concentrate contributes to the beneficial effect of the finished form of fertilizer on the complex of soil microorganisms possessing economic-useful traits, which significantly increases the value of fertilizer.

Dissolution of salts used as mineral fertilizers in whey hydrolyzate allows to obtain a wide range of fertilizers adapted for different cultures and different types of soils. The mineral complex of salts allows not only to vary properties and purpose of fertilizers, but also provides a preservative effect and stability of the agricultural product during storage.

To isolate dry components from the low concentration whey fraction, a sorption method can be used. The use of glauconite concentrate as a sorbent possessing a complex of valuable agrochemical properties makes it possible to use the spent sorbent as an effective organomineral fertilizer for local application to the soil or as an additive to artificial soils.

The proposed technological scheme of whey processing can be implemented at milk processing plants of small and medium capacity. The introduction of the technology of whey processing into fertilizer will allow to significantly reduce the pollution of the environment by the enterprises of the dairy industry and to obtain additional income.

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