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Treatment of Lake Waters by Titanium Dioxide and Clay as A Flocculating Agent: Case of Lake Antagnavo, Antsiranana Ii District, Madagascar.

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Abstract - The main objective was to assess the treatment efficiency of a clay and TiO2 based system for removing pollution from lake waters, specifically Lake Antagnavo. Analysis of physicochemical parameters (pH, conductivity, suspended solids, turbidity, nitrite, nitrate, phosphate, and chloride levels, COD, and BOD) and bacteriological parameters (ASR, fecal coliforms, fecal streptococci, and Escherichia coli) allowed for the evaluation of the treated water quality. The model reduced the amount of physicochemical pollution in the wastewater at the end of treatment. The pH ranged from 6 to 9, the conductivity decreased from 429.33 to 273 µS/cm, the turbidity was 17.8 NTU, and the suspended solids varied from 12.5 mg/L. Nitrite and phosphate levels are 0 mg/l. Nitrate and chloride levels are 11.82 mg/l and 26.08 mg/l, respectively. BOD5 is less than 50 mg/l, and COD is less than 150 mg/l; all values comply with discharge standards. According to these results, 99.78% of fecal streptococci, 99.64% of ASR, 97.84% of fecal coliforms, and 98.43% of Escherichia coli are eliminated. Microbiological and physicochemical parameters reveal that the treated water is qualitatively improved.

Keywords - Lake water, Pollution, treatment, Titanium dioxide, Clay.

I. INTRODUCTION

Environmental degradation, and its direct consequences, such as climate change and the extinction of certain species, are all phenomena that concern and challenge us right now. These phenomena are primarily due to human activities: population growth, rural development, and overproduction in all sectors of the economy, particularly in agriculture. These human activities consume and discharge large quantities of water. Lake water, among other things, is one of the main factors in environmental degradation of domestic or agricultural origin; it flows into sewers, streams, and rivers, and is discharged into seas and oceans, carrying toxic products used upstream and harming aquatic flora and fauna. Faced with this problem, we attempted to treat these lake waters using a clay and TiO2-based model. Our study is entitled "Trial of lake water treatment using titanium dioxide and clay as a flocculant agent: the case of Lake Antagnavo." The objective of these treatments is to reduce the impact of lake waters before their discharge into the environment, to degrade pollutants, and to reduce pathogens in the lake water. The use of a clay and TiO2-based model is beneficial from both an environmental and human health perspective [9]. Titanium dioxide is one of the most basic materials in our everyday lives [10]. It is used in a wide variety of paints, cosmetics, and food products. Today, global annual consumption of TiO2 exceeds three million tons [11]. In other cases, the photoactivity of TiO2 plays a positive role: for example, in the degradation of malodorous or irritating chemicals [12], toxic products, and bacteria. For all these applications, photocatalytic technology can be the subject of industrial development. Clay plays an important role in certain fields, such as pharmaceutical manufacturing and the treatment of polluted water, for example, in the adsorption of toxic organic compounds [13]. The analyses were carried out by the laboratory of the Research Unit in Process Engineering and Environmental Engineering (URGPE) and the Laboratory of Environmental Microbiology (LME).

II. MATERIALS AND METHODS

Biological Materials for Study
Type of Water Studied

Lake Antagnavo is located 75 km south of the city of Antsiranana and 4 km east of the town of Anivorano-Nord in the DIANA region of northern Madagascar. Geographic coordinates: 12°45′00′′ S and 49°15′00′′ E. Type: crater lake, Surface area: 1.56 km^2, Perimeter: 4,704 m, and Altitude: 420 m.

Figure 1: Lake Antagnavo

Reagents Used

The reagents used are: clay, titanium dioxide (TiO_2) powder, wastewater, and hydrochloric acid.

Equipment Used During Treatment Testing

- Sand filter: to remove suspended solids and obtain a clear solution.
- Sterilized bottle: to prevent contamination of the sample.
- Oven: to dry the clay
- Balance
- Beaker

Media for the Detection and Enumeration of Germs in Lake Water

- TTC and Tergitol 7 Lactose Agar : This is a selective and differential medium that allows the enumeration of coliforms and Escherichia coli in water samples.
- Enterococcus Agar : This is a selective solid medium that allows the isolation and enumeration of fecal Streptococci by membrane filtration.
- TSC (Tryptose Sulfite Cycloserine Agar) : This is a selective and differential medium for the enumeration and presumptive identification of Clostridium perfringens.

Methods
Principle

It consists of treating all organic matter and microorganisms (ASR, fecal coliforms, fecal streptococci, E-coli) in raw water by the treatment process (TiO_2 + Clay).

Procedure

- Preparation of titanium dioxide:
- Mold the clay into a solid disc
- Dry it in an oven at a minimum of 100°C
- Activate it with concentrated hydrochloric acid for a few minutes
- Weigh out some titanium dioxide powder
- Dissolve the TiO_2 in hydrochloric acid until completely dissolved
- Apply the solution to the pores of the prepared clay
- Dry in an oven at approximately 300°C for 1 hour.

Treatment preparation:

Filter the sample through a filter (sand or paper) to remove suspended solids.

Place the filtered raw water in a beaker.
Place the treatment preparation at the bottom of the beaker for a few minutes in the presence of light.

Test aliquot:

Mass of titanium dioxide = 0.2188 g.
Volume of HCl = 30 mL (for dissolution).
Mass of clay = 11.33 g.
Volume of raw sample = 300 mL.

Parameters to be analyzed
Physical parameters: temperature, pH, electrical conductivity, suspended solids, turbidity.

Chemical parameters: nitrate, nitrite, chloride, and phosphate levels. Biological parameters: diochemical oxygen demand (BOD) and chemical oxygen demand (COD). Microbiological parameters: detection and enumeration of spores of sulfite-reducing anaerobic bacteria, fecal streptococci, fecal coliforms, and Escherichia coli.

Physicochemical Parameters - pH
The pH is measured using the standardized method NF-T 90-008. It can be performed using a pH meter with an electrometric method and a glass electrode. Measurements are expressed in pH units at a temperature of 25.1°C. The pH of the discharge must be between 7.26 and 7.66. The further an effluent deviates from neutral pH (pH=7), the more difficult it is for biological life.

Temperature
The temperature is measured using a liquid thermometer, accurate to within ±0.1°C. Temperature plays a crucial role in physicochemical analysis and must be considered when determining the impact of domestic and industrial discharges.

Conductivity
Electrical conductivity is a measure of the current conducted by ions present in water and depends on the ion concentration and the nature of the ions. It provides an assessment of the dissolved salt content of wastewater. It is measured by an induction-type conductivity meter (NF ISO 7888 – 1985).

Turbidity
Wastewater turbidity is defined as the expression of the optical property whereby light is dispersed and absorbed rather than transmitted in a straight line through the sample. This dispersion is caused by the interaction of light with suspended particles and reflects the reduced transparency of the medium due to the presence of these particles. It is generally measured in nephelometry by comparing the light transmitted, on the one hand, by the sample being tested, and on the other hand, by a standard curve consisting of a formazine solution, under incident radiation of a given wavelength (NF-T 90-033 standard).

Suspended Solids (SS)
A representative sample is filtered through a paper filter membrane that has been previously rinsed and dried at 1050°C. The solids retained on the filter are dried and then weighed with the filter. The mass of suspended solids is obtained by subtracting the initial mass of the filter, taking into account the filtered volume, from the mass of the filter including the dried residues. When a filter membrane is used, all residues passing through the pores of the filter are considered soluble. Therefore, any colloidal material small enough to pass through the filter appears soluble. The collected sample is filtered using a standard filtration device; the filter is then dried in an oven, and the mass of the residue retained by the filter is measured with a precision balance. We used the vacuum filtration method on a paper filter disc.

Nitrates
In the presence of sodium salicylate, nitrates form sodium paranitrosalicylate, a yellow-colored compound that can be determined colorimetrically. The measurement is performed by UV-visible spectrophotometry at a wavelength of 415 nm. A blank test is required for this measurement.

Nitrites
In the nitrogen cycle, nitrite ions are relatively transient intermediates between ammonia nitrogen and nitrate ions. High nitrite concentrations in the ocean environment are possible due to several types of phenomena.

Phosphates
Phosphates are compounds that contain phosphorus. The phosphate content in the sample is determined by UV-visible spectrophotometry at a wavelength of 880 nm (NF-T-90-023).

Chloride
Chlorides are titrated in a neutral medium with a standardized ammonium thiocyanate solution. The end of the reaction is indicated by the appearance of

a pink color. Expression of results: For a 50 mL test aliquot, the quantity of chloride, expressed in mg/L.

Biochemical Oxygen Demand (BOD5)

In this procedure, the manometer method is used, based on the principle of the Warburg respirometer, in which biomass respiration is directly measured by an instrument. The BOD5 measurement depends on the activity of the microorganisms present in the water sample. The standardized method uses an oximeter: an electrical device equipped with a probe that provides an instantaneous measurement of the oxygen level in the water. By measuring the oxygen level in an air-free test tube on the day of preparation and after 5 days of culture, it is possible to determine the proportion of oxygen consumed by the microorganisms for their metabolic needs over this period.

For the OXYTOP method, test tubes are filled with a predetermined sample volume. A sodium hydroxide pellet is placed in the test tube to absorb the CO2 produced during the oxidation of organic matter by bacteria. The test tube is hermetically sealed with a special stopper equipped with a digital pressure gauge with memory, allowing for the measurement of the pressure drop that gradually develops as the CO2 is absorbed. By correlating this with a scale, the BOD5 of the sample can be determined.

Chemical Oxygen Demand (COD)

To estimate the amount of oxygen required for a given sample to be oxidized, the standard method involves adding potassium dichromate to allow the oxidation of all susceptible organic or mineral materials. This reaction is carried out in an acidic medium, and the mixture is brought to a boil in the presence of a catalyst (AgSO4). After this reaction, the remaining dichromate in solution is titrated, and the amount of oxygen required by the reaction is estimated by the difference. This titration is performed by reducing the remaining dichromate with a solution of Mohr's salt [FeSO4, (NH4), 2SO4, 6H2O]. Ferroin, a colored indicator, changes from blue to red when this reaction is complete.

Note: Not all compounds are oxidized during this reaction; some complex molecules remain unchanged. In the case of typical rural wastewater, this has little impact on the measurement.

Analysis of Bacteriological Parameters

Preparation of Wastewater Sample for Analysis

One hundred (100) milliliters of the water to be analyzed, or 100 ml of its decimal dilutions, are filtered through a sterile membrane with a porosity of 0.45 µm and a diameter of 47 mm. Dilution involves mixing precise quantities of the sample with sterile distilled water until a suitable concentration is obtained for enumerating microorganisms. The dilutions used (generally from 10^{-1} to 10^{-3}) vary depending on the microorganism, and three replicates were performed for each dilution.

Detection and enumeration of studied bacteria

Detection and enumeration of fecal streptococci (ISO 7899-2:2000): The membrane is placed on a pre-poured and solidified m-Enterococcus agar medium. The cultures are then incubated at 37°C for 48 hours. After incubation, the purple or pink colonies are counted. The confirmatory test was performed by examining the microscopic appearance (Gram-positive cocci, spherical or ovoid, forming chains) and the biochemical characteristics (catalase negative).

Detection and enumeration of fecal coliforms (ISO 9308-1): The membrane is placed in a Petri dish containing TTC and Tergitol 7 Lactose Agar medium. After 18 to 24 hours of incubation at 37°C, the characteristic yellow to orange color colonies, with the presence of a yellow halo in the mass of the medium under the membrane, are enumerated.

Detection and enumeration of Escherichia coli (ISO 9308-1): The genus Escherichia belongs to the fecal coliforms and is the only one that is unequivocally always of fecal origin. After filtering the sample, the membrane is placed on TTC and Tergitol 7 Lactose Agar. This allows coliform colonies to grow during an incubation period of 18 to 24 hours at 37°C. Colonies considered lactose-positive are those on the underside of the membrane, which are yellow. Typical colonies are subcultured onto TSC agar and incubated at 37°C for 24 hours and in tryptophan broth incubated at 44°C for 24 hours. Oxidase 6 and indole-positive colonies are considered Escherichia coli.

Detection and Enumeration of Sulfite-Reducing Anaerobic Bacteria Spores (NF T 90 415): The water sample is heated to 80°C for 10 minutes and then subjected to thermal shock by being immersed in cold water. The sample (100 ml) is filtered through a membrane filter and placed with the upper surface facing down in a Petri dish. Supercooled TSC culture medium is then added, and the spore count is performed after incubation at 37°C for 24 hours under anaerobic conditions. Characteristic colonies are black.

Experimental Results

To determine the quality of lake water, physicochemical and bacteriological analyses are mandatory.

Results of Physicochemical Analyses

The table below shows the results obtained during the analysis of the physicochemical parameters of the sample.

- Physical parameters: temperature, pH, conductivity, turbidity ;
- Chemical parameters: chloride, nitrate, nitrite, phosphate.

Table 1: Physico-chemical qualities

Settings	E1	E2	E3	AVERAGE	E 4	SMS
pH	7,66	7,46	7,30	7,47	6,62	6 à 9
TSS (mg/l)	153	100	110	121	12,5	< 70
Turbidity (NTU)	59	45,1	60,9	55	17,8	< 20
Conductivity (µs/cm)	495	476	317	429,33	273	< 1500
Temperature (°C)	25,1	25,1	25,1	25,1	25,1	< 30
Nitrate (mg/l)	14,03	12,004	13,384	12,806	11,82	< 20
Nitrite (mg/l)	0	0	0	0	0	< 0,2
Phosphate (mg/l)	0	0	0	0	0	< 10
Chloride (mg/l)	39,08	23,156	29,804	30,68	26,08	< 250
DBO5 (mg/l)	70	80	80	76,67	46	< 50
DCO (mg/l)	156,42	170,72	163,18	163,44	139,014	< 150

SMS : Standard MALAGASY STATE

E4: Sample after treatment

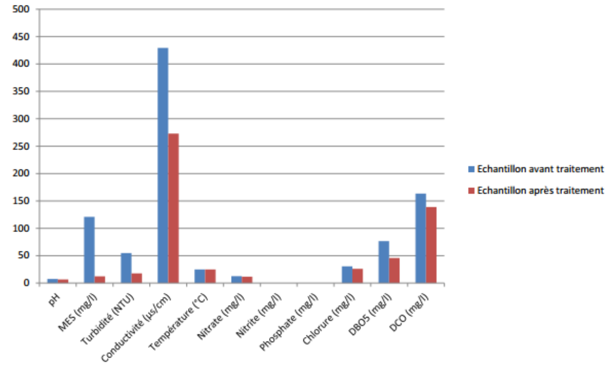

Figure 2: Evolution of physico-chemical parameters.

The variation in different parameters determines the effectiveness of the implemented process. According to the results, the average suspended solids (SS) value of the raw water analyzed exceeded the discharge water standards (< 60 mg/l), indicating a high concentration. It is therefore very rich in dissolved and colloidal matter. The turbidity of the samples taken is significantly higher than the standard, reaching up to 80 NTU. This indicates that the raw water contains a high level of colloidal matter, as turbidity is directly related to the determination of suspended solids. The average pH value of this raw water is around 7.47.

It complies with the WHO standard (6 < pH < 9). Nitrates and nitrites pose a danger to fauna and flora when present in excessive quantities. The nitrate and nitrite concentrations in our raw samples did not exceed the required quality standard. Compared to the standard for lake water discharges (10 mg/L of phosphate), phosphates do not represent a significant pollution factor in the sampled lake waters. The amount of phosphates at the sites is less than 5 mg/L.

High chloride concentrations lead to water toxicity. Their presence is dangerous for vegetation, yet the results show that chloride concentrations in lake waters are low compared to discharge standards. The concentration of organic matter is determined by BOD5 and COD analyses.
COD is always higher than BOD5 because many substances can be chemically oxidized but cannot be biologically oxidized. This indicates that lake waters are biodegradable and contain microorganisms. The results of our analyses confirm the presence of a large quantity of organic matter in the wastewater.

After treatment, an improvement is observed in almost all parameters such as pH, turbidity, suspended solids, chloride level, phosphate level, nitrate level, nitrite level, COD and BOD5 levels; that is to say, all physicochemical parameter values are reduced. The values comply with wastewater discharge standards.

Microbiological Quality of Lake Waters Before and After Treatment

Analysis Before Treatment

The results obtained during the microbiological analyses of wastewater E1 are presented in the following table. These results were provided by the CNRE's specialized laboratory. According to the table below, the microorganism count in sample E1 is significantly higher than the standards, meaning that the analyzed wastewater is highly polluted. The microbiological quality of the lake waters before treatment is as follows.

Table 2: Microbiological quality of lake waters before treatment (E1 lake waters before treatment)

Enumeration	E2	Unit	Methods	Criteria
ASR	$2,5.10^2$	UFC/100 ml	ISO 6461- 2 :1986	100
Fecal coliforms	67	UFC/100 ml	ISO 9308- 1 :2000	500
Fecal streptococci	12	UFC/100 ml	ISO 7899- 2 :2000	100
Escherichia coli	94	UFC/100 ml	ISO 9308- 1 :2000	100

The values of the microbiological characteristics of the wastewater before and after treatment are presented in Tables 2 and 3. These results were provided by specific laboratories. The figures below show the enumeration rates of the microbiological parameters and the differences between the E1 and E2 samples analyzed, compared to the standards.

Enumeration of ASR (Anaerobic Sulfite-Reducing)

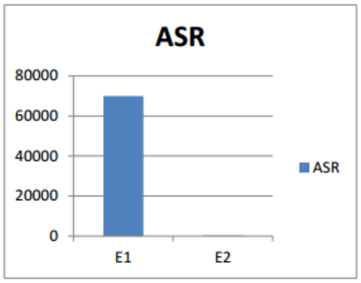

Figure 3: Difference between treated wastewater E1 and E2

According to this histogram, there is a significant difference between raw lake water E1 and treated wastewater E2. The number of sulfite-reducing anaerobic bacteria (SRB) colonies in wastewater E2 is much lower than in E1. This value after treatment is below the required standard (WHO discharge standard). Indeed, the treatment process progressively destroys microorganisms. The reduction rate is 99.64%, i.e., 70,000 CFU/100 ml, and becomes 250 CFU/100 ml after treatment.

Fecal coliform count:

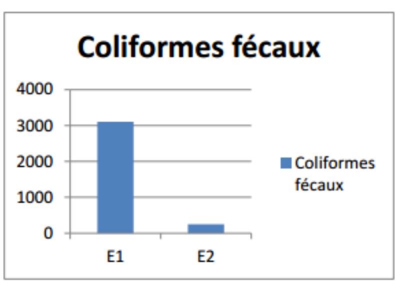

Figure 4: Difference between treated wastewater E1 and E2

The presence of fecal coliforms indicates the presence of a fecal source (manure, septic tank, or other) polluting the water. According to the results, the same phenomenon was observed with fecal coliforms; the number of colonies in wastewater E1 is higher compared to treated wastewater E2. This significant decrease in the number of colonies is due to the effectiveness of the treatment process (clay-TiO_2). The colony count in E2 is below the standard. The count rate is 97.84 %.

Fecal streptococcal count:

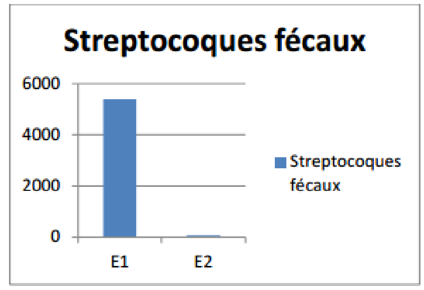

Figure 5: Difference between treated wastewater E1 and E2

Fecal streptococci are also a source of fecal pollution in water. According to the analysis, fecal streptococci are present in samples E1 and E2. The number of fecal streptococcal colonies decreased after treatment, from 5400 CFU/100 ml in the raw water to 12 CFU/100 ml after treatment. The count rate was 99.78 %.

Escherichia coli count:

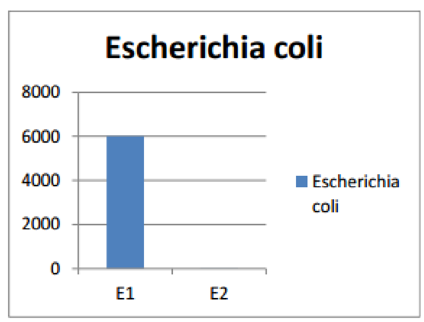

Figure 6: Difference between treated wastewater samples E1 and E2

Based on the results, the decrease in the number of Escherichia coli colonies observed in the treated wastewater sample E2 is highlighted in the count curve (Figure 6). Furthermore, the decrease in the number of microorganisms indicates the degradation of the bacteria in the treated water. The reduction rate is 98.43%, decreasing from 6,000 CFU/100 ml to 94 CFU/100 ml, a value that meets the standard.

Discussion

Each data point in this table represents the mean obtained from a small number (less than 10). Analytical studies show that lake waters exhibit high levels of toxic contamination, whether organic, mineral, metallic, or bacteriological. The results highlight wastewater pollution. According to our analysis, lake waters contain a wide variety of microorganisms, some of which can promote the decomposition of organic matter and the recycling of essential nutrients for the maintenance of aquatic organisms and the food web [4].

However, other microorganisms originate from animal and human waste and can cause serious illnesses in humans, such as gastroenteritis and skin infections [6]. Indicator bacteria found in large numbers in the digestive tract of warm-blooded animals, such as fecal coliforms (thermotolerant coliforms) and Escherichia coli (E. coli), are used to assess the level of bacterial contamination in water [5]. Other potentially pathogenic germs (staphylococci, Escherichia coli, intestinal enterococci, etc.) may coexist with the identified parasites, which will have a direct impact on the health of users, especially the youngest and oldest among them.

Indeed, the consumption of foodstuffs, raw vegetables, and the use of water contaminated by these microorganisms could increase the already rich and varied intestinal flora in humans, potentially leading to metabolic imbalances [2] and serious health risks. To preserve the ecosystem and avoid the risks to public health posed by lake water pollution, comprehensive treatment of lake water is mandatory. To prevent harmful effects, all the legislation we have mentioned aims to control water quality and regulate treatment, all with the goal of protecting human health and the environment [8]. However, treating or preventing human or animal diseases can have downstream effects [1].

Furthermore, the harmful effects of lake water also pose a danger to food quality and human health, as demonstrated by several studies [3]. The detection of these bacteria can indicate the presence of other microorganisms, such as bacteria, viruses, and protozoa, which can cause illness, the most common being gastroenteritis [7]. Although often mild, it can sometimes have serious health consequences. Those most likely to be affected are children under 5, the elderly, and people with weakened immune systems or chronic illnesses.

Research has demonstrated that the bacteriological quality of these treated lake waters is acceptable and meets WHO standards. Our work, conducted over a specific period, aimed to determine the standard for bacteriological quality. Based on the results, we observed that the solution is nearly reliable; that is, after treatment, the levels of bacteria (ASR, Escherichia coli, fecal streptococci, and fecal coliforms) are progressively reduced. This indicates a change in water quality. This improvement demonstrates an elimination or reduction in the number of pathogenic strains in the sample. The outcome of our work has highlighted the importance of using the treatment process (clay with TiO_2): it facilitates the elimination of certain pathogenic bacteria such as ASR, Escherichia coli, fecal streptococci, and fecal coliforms with a 99% efficiency rate.

Recommendation

Bacteria are microorganisms that can break down and assimilate a large portion of the organic matter contained in wastewater. These bacteria release degradation products into the environment, which are soluble minerals and dissolved gases. Depending on the balance of the environment, and in particular the nitrogen and phosphorus levels, the best-adapted bacteria develop rapidly and dominate other species. A natural regulation of bacterial levels is observed, based on the organic matter present in the environment and other growth conditions (pH, dissolved oxygen, etc.). That is to say, when the level of organic matter in the water increases, the bacterial level also increases, and vice versa.

III. CONCLUSION

Lake waters are particularly favorable environments for the development of microorganisms of all kinds, especially pathogenic bacteria and viruses. The microbiological composition of wastewater is highly influenced by lifestyles and regional sanitation conditions. This study, based on the removal of bacteria and organic matter from lake waters, allowed us to demonstrate the effectiveness of the clay-titanium dioxide process in degrading pollution in raw water. The decrease in the value of each parameter studied is evidence of this. We observed that the colony counts of each microorganism in the treated water were below the standards for wastewater discharge.

We can demonstrate the important role of the treatment process on the microbiological quality of lake waters. Indeed, it reduces the number of bacteria present in this wastewater by 99%. Our experimental study is limited to treatment based on the characteristics of the wastewater. The products are difficult to prepare and their cost is high compared to conventional products. Improving physicochemical analyses and studying the economics of using the product could be the subject of further research. The treatment of lake waters has only one objective: environmental protection. It is part of a set of measures to combat environmental degradation: it contributes to the protection of plant and animal species.

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