



Aerobic stabilization of organic waste effluents from anaerobic treatment for agricultural use

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Received 29 October 2019; Accepted 10 March 2020

ABSTRACT

Anaerobic bioconversion of organic wastes to retrieve energy and industrially usable compounds has been studied for long. Although anaerobic effluents retain (i) humifiable, organically bound carbon, and (ii) and agriculturally important macro- and micro-nutrients, their agricultural utilization is limited due partly to environmental legal regulations, partly to agronomic, soil ecological, and plant physiological concerns. The present review considers stabilization methods for organic effluents that conserve materials of agricultural importance, available in them while satisfying environmental protection thresholds for the cleaned effluents; and are simply feasible, easy to design, practical to fit in existing sewer technology, and cost-effective to implement. The review intends to identify the most promising research pathways to stabilize anaerobic effluents, with the specific aim of utilization in agriculture. Methods considered include augmented biofiltration and composting.

Keywords: Aerobic stabilization; Anaerobic effluent; Bio-electrochemical system; Biofilter; Composting; Digestate; Humification; Nutrient recovery; Organic waste

1. Introduction

According to FAOSTAT searchable online database, the agricultural sector of the European Union used 11,350,000 tons of nitrogen, 2,663,000 tons of P_2O_5 , and 2,972,000 tons of K_2O fertilizer in 2015 [1]. During the same period, the EU produced 18 billion m^3 of methane equivalent in 17,439 biogas plants [2], emitting digestate containing cca. 44 Tg (million tons) of dry matter [3]. This may have contained up to 1.85 Tg of nitrogen, 0.7 Tg of P_2O_5 , and 0.38 Tg of K_2O active matter. Only by using the digestate from industrial and agricultural biogas plants, EU agriculture could have saved 17% of the total mixed fertilizer (active substance) in 2015. These figures do not even include the effluents of municipal sewage sludge digestors, because those are re-fed to the main treatment stream.

More importantly, this amount of digestate contained about 22 Tg of organically bound carbon. In anaerobic effluents, nearly 15% of this carbon is bound in stable organic substances. In addition, 25%–30% of the biodegradable part can be humified within a year under natural conditions, or within months by composting. To quantify the significance of these quantities, this more than 8 Tg of stable organic carbon could compensate for most of the missing humus content on all cultivated land in Hungary, lost due to decades of industrialized agriculture. For comparison, global loss of soil organic carbon content is over 1.6 Pg/y from the upper 100 cm layer [4].

BOD removal efficiency of anaerobic processes never reaches 100%, effluents contain residual organic matter that is biodegradable under aerobic conditions. They are

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Presented at PERMEA 2019, 26–29 August 2019, Budapest, Hungary

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therefore biochemically unstable. As soon as exposed to air and light, spontaneous – both aerobic and additional anaerobic – microbial activity starts in these liquids, as detailed in section 3.3 “Stabilization through nutrient recovery.” As such, these liquids cannot be discharged to the natural ecosystem. Agriculture needs valuable substances that these effluents retain, but their direct land application is limited. The rationale of the present review is directly deduced.

Innovative aerobic stabilization methods are to be studied that:

- recover agriculturally important substances from anaerobic effluents, while
- cleaning the remaining liquid phase to attain environmental protection thresholds for discharge.

2. Anaerobic effluents

Organic wastes can be converted to energy as well as industrially usable compounds by anaerobic processes, such as methanogenesis, hydrogen fermentation, microbial fuel cells, microbial electrolysis, etc. Irrespective of the anaerobic process used, the effluent retains agriculturally important macro and micronutrients [5], and – what nowadays agriculture is missing the most – humifiable, organically bound carbon [6,7].

2.1. Biogas digestates

Biogas digestate is a classified waste in the European Union, its discharge is subject to severe regulation in Hungary, as well. Each delivery requires specific permission, based on laboratory analysis. According to the relevant

literature, the composition of a few typical biogas digestates are summarized in Table 1.

Liquids of such composition cannot be released to the natural ecosystem, as they have components at levels far above the emission thresholds. For comparison, Table 2 presents some typical water quality standards under Hungarian environmental legislation.

Direct land application of anaerobic effluents is conditionally authorized, on a case by case basis, depending on the geological characteristics of the site, the cropping, the season, and the nutrient content of the soil. Application is, however, largely limited due partly to legal regulation (EU nitrate directive, Natura 2000 rules, and environmental emission thresholds), partly to agronomical and biological concerns detailed in section 3 (Stabilization methods). Instability of biogas digestates is due to their high nitrogen, phosphorus, and organic carbon content, because

- these are the principal nutrients for subsequent aerobic microbial activity,
- ammonia and VOC volatilization,
- uncontrolled spontaneous denitrification – nitrification cycles that contact with the air triggers.

Available analysis data is incomplete and vary according to raw substrate composition and technology [11–13]. They do however show that:

- the digestates are moderately basic with pH 7.5–8.5,
- nitrogen and phosphorus concentration against remaining carbon content exceed physiological needs, with a C/N ratio of 4–9, instead of 15,
- nitrogen is present mostly in $\text{NH}_4\text{-N}$ form.

Table 1
Typical digestate compositions

Parameter	[8]	[9]	[10]	[4]	
				Holsworthy	Ribe
DM, %	7.7			5.8	5.8
C/N	7.0				
pH	8.2	8.2			
TN, kg/m ³	5.0		1.8% DM	6.6	4.9
NH ₄ -N, kg/m ³	2.8	0.13		5.0	3.2
NO ₃ -N, kg/m ³		0.02			
PO ₄ -P, kg/m ³		0.004			
P ₂ O ₅ , kg/m ³			0.8% DM	3.3	2.4
K, kg/m ³	4.9				
K ₂ O, kg/m ³			0.75% DM	4.5	4.2
MgO, kg/m ³	0.95				
CaO, kg/m ³	2.2				
COD, mg/L		820			
Chlorides, mg/L		369.5			
EC, μS/cm		2,318			
Total dissolved solids, ppm		1,159			
Salinity, PSU		2.7			

Table 2
Environmental thresholds in Hungary

Good water quality for surface water/ Decree 10/2010. (VIII. 18.) VM	Technical emission thresholds for surface water/ Decree No. 28/2004. (XII. 25.) KvVM
pH: 6.5–9	pH: 6–9.5
EC: <1,000 $\mu\text{S}/\text{cm}$	
Dissolved oxygen: >6 mg/L	
BOD ₅ : <4 mg/L	BOD ₅ : <50 mg/L
COD _{cr} : <30 mg/L	COD: <150 mg/L
TN: <3 mg/L	TN: <55 mg/L
TP: <500 mg/m ³	TP: <10 mg/L
	VS: <200 mg/L

The authorities may prescribe stricter thresholds for the applicant if necessary, for maintaining good water quality.

This is understandable, since methanogenic bacteria are active in a slightly basic environment, in this case provided by the presence of ammonium–nitrogen, and a part of the initial biodegradable carbon content has been released in the form of CH₄ and CO₂ gases.

2.2. Effluents of other anaerobic bioprocesses

The literature on other bioprocesses, such as anaerobic fermentation or bio-electrochemical systems (BES) is abundant. Far less information is available on the composition of their effluents, and even the available information is incomplete and hardly usable for comparison. Most experiments on BES use artificial substrates composed only of the few substances to be studied. Even those experiments where real substrates are used, only monitor those parameters that are essential for the purpose of the particular tests in question. For illustration, Table 3 shows analysis results of biohydrogen dark fermentation and BES effluents, published in the relevant literature. Organic matter and electrochemical parameters are usually covered, often in high resolution. Chemical oxygen demand (COD), biochemical oxygen demand (BOD₅), SS, NH₄–N, pH, EC values appear sporadically, but never the full scope within the same study.

In contrast to biogas digestate, the pH of dark fermentation effluents are in the acid zone, and oxidized forms dominate the N-content instead of NH₄–N. Substrate composition may account for this difference: biohydrogen is usually produced from vegetal raw materials of wide C/N ratio. These are rich in polysaccharides, such as starch, pectin and cellulose, and relatively poor in amino acids.

The whole field of BES remains firmly in the research phase, the practical application does not seem to have reached the point where environmental effects, agricultural or other use of BES effluents would be studied. But the BES process is globally analogous to biogas fermentation with anaerobic oxidation of organic matter delivering energy in both processes. The main difference is the form this energy is delivered: hydrogen or electric current, rather than methane. Then, it is not unreasonable to suppose that BES effluents are subject to similar changes, compared to the fed substrate:

- total carbon content decreases,
- ratio of stable organic carbon within the total carbon content increases,
- ratio of nitrogen and phosphorus to carbon extends,
- pH increases.

3. Stabilization methods

Anaerobic effluents are still unstable, therefore they cannot be emitted to the natural ecosystem, and their storage is expensive. A wide range of stabilization methods has been created to treat liquid organic wastes in the past decade, and further research in this field continues due to stricter regulation and cost issues. Most of the studied methods, however, offer an only partial solution or are unreasonably expensive.

3.1. Direct land application

At first glance, the most obvious way to stabilize anaerobic effluents seems to be bioconversion in the rhizosphere of crops on cultivated land. The major disadvantage of this method is that in a moderate climate, the available time frame is insufficient to apply the whole annual quantity at reasonable cost. For instance, depending on seasonal fluctuations and geographical factors, only 2–3 months are available for direct land application of anaerobic effluents in Hungary. This is due to legal, soil ecological, agronomical, and plant physiological constraints, including:

- intermittent anaerobic conditions in the rhizosphere harm soil ecosystem,
- temporary increase of salinity in the topsoil slowing plant development in the most sensitive phenophase,
- high application rates damaging soil structure,
- the timing of authorized application not corresponding to the nutrition and water need of the crop in the actual phenophase,
- the proportion of nutrients present in the effluent not corresponding to the need of the crop,
- depending on the effluent, pathogen microbes being released,

Table 3
Analyses of non-methanogenic bioprocesses in the literature

Parameter	[14]	[15]	[16]
	Biohydrogen dark fermentation effluent	Dark fermentation effluent treated in MFC	Biogas digestate treated in MFC
pH	4.53		6.8–7.2
EC, mS/cm			0.91
TS, g/L	34.9		9.2
VS, g/L	21.6		7.8
TCOD, g/L	41.5		4.9
VCOD, g/L			0.8
Total VFA, mg/L		1,634.77 (8 substances)	
TN, mg/L	686		
TP, mg/L	300		
Total sugars, mg/L		425.20 (5 substances)	

- under moderate climate cereals – the most cultivated crops – usually receive sufficient natural water supply during their phenophase of rapid growth; land application of effluents may lead to over-irrigation.

In addition to these factors, slurry from intensive animal husbandry and other wastewaters compete for this narrow time window. Moreover, NH_3 volatilization leads to at least 30% nitrogen loss even when using the best available practices. Paradoxically, land application of the whole annually available quantity of this – in principle – much needed material would cause more harm than use, under moderate continental climate.

3.2. Stabilization with wastewater treatment methods

Anaerobic effluents can also be stabilized with usual methods of wastewater treatment, as is the current practice at, for example, sewage sludge digestors. The essence of these methods is the exact opposite of agricultural utilization of anaerobic effluents: it is to transform “polluting” organic carbon and nitrogenous compounds from the substrate to CO_2 and inert N_2 gas, releasing them to the atmosphere. They also bind phosphorus through precipitation in insoluble compounds. On short term, this practice appears environmentally sound. But these same substances, retrieved with cost and energy spending, have to be re-synthesized in (again) costly and energy-intensive fertilizer factories for agriculture. Therefore, in practice, the application of wastewater treatment methods does not fully mitigate water pollution. Instead, they convert a part of it to air pollution – at substantial expenses. Given this, it is surprising that a large part of relevant contemporary scientific literature in this area continues to cover such problem-shifting research.

3.3. Stabilization through nutrient recovery

The biological instability of anaerobic effluents is caused by their residual nitrogen, phosphorus, and organic carbon content. Several microbial processes start when the

effluent comes in contact with the atmosphere. Organic matter supplies nutrients to the metabolism of heterotrophic microorganisms (using atmospheric O_2). In addition to this, the two eutrophication substances, $\text{NH}_4\text{-N}$ and phosphorus, nourish photoautotrophic activity (using atmospheric CO_2) in the presence of sunlight. Furthermore, NO_x -nitrogen content provokes spontaneous denitrification – nitrification cycles, leading to nitrogen loss.

These same substances are the most important input materials for plant cultivation. An emerging part of scientific research aims at the long term, general solution to the biological stability: nutrient recovery. Among these, the following methods stand out:

- crystallization of joint salts of ammonium and phosphate ions (NH_4MgPO_4 – struvite, and $\text{NH}_4\text{H}_2\text{PO}_4$ – MAP) [17],
- nutrient concentration with BES [18,19],
- binding organic carbon and NPK content in vegetal biomass, then in stable organic macromolecules – completed with photosynthetic CO_2 binding. The process may be implemented in:
 - algal photo-bioreactor [20–22],
 - vegetal rhizosphere [23–25],
 - cathode cell of microbial fuel cell supplied with photosynthetic biocathode, be it operated either by algal [26,27], or vegetal culture [28–30].
- binding organic carbon and NPK content in humus precursors through microbial conversion (biofiltration, composting, or both).

Deficiencies of such innovative processes are:

- nutrient crystallization does not treat substrate carbon content which, today, is the most missing substance for agriculture;
- only 16%–18% of total phosphorus content is present in dissolved orthophosphate form, therefore only a small, stoichiometrically proportional part of ammonium–nitrogen content can be bound to [11]; this is hardly more than 1% of the available $\text{NH}_4\text{-N}$ in municipal sewage sludge digestate, for example;

- the process is efficient at a pH above 9, exceeding both substrate pH and the environmental emission threshold, which would entrain an artificial intervention both ways;
- BES treat only one, exceptionally two of the macronutrients – at the expense of organic carbon content;
- nutrient recovery through biomass requires post-treatment (e.g., humification by composting) in order to obtain a stable final product.

The most obvious way to apply stable organic carbon substances, that is, humus forming matter on agricultural land is compost fertilization. Among the above-reviewed methods, biological nutrient recovery – with photosynthetic carbon supplementation or through direct microbial conversion – seems the ecologically soundest and most economically feasible choice. As the end product of such processes is compost, it fits the optimal land application method. As such, bioconversion methods of anaerobic effluents are reviewed in more detail in the following section.

4. Microbiological methods of nutrient recovery

4.1. Biofiltration

Upon biofiltration, microorganisms (or rhizosphere organisms, in the case of constructed wetlands) take up the targeted pollutants. The microorganisms grow on a matrix material. The target substance is partly used for cell construction, partly transformed, and returned to the substrate as intermediate metabolites, and partly serves as an energy source for the first two processes. Although the matrix physically filters suspended solids, too, this is only a collateral advantage, secondary to the main process of bioconversion.

Microbes in the biofilter can be fixed both to inorganic and organic matrices. Inert materials with high specific surface area and pore volume (of over 40%) are usually chosen, that are locally available and inexpensive. The inorganic materials most commonly utilized are sand and gravel, while slowly biodegradable vegetal parts, such as almond shell, coir, stems, and leaves of different reed species are usually chosen as the basis of organic matrices [31]. Ceramic beads and plastic carriers – borrowed from moving bed biofilm reactor technology – may equally be used, although these are more expensive. Mineral wool was studied as a possible anaerobic biofilter matrix in the 1990s [32], for recirculating irrigation water in horticulture. In the study, a 0.6 m deep rockwool filter entirely removed *Phytophthora* spores from recirculated water at a surface flow rate of 81.4–122.1 L/d, depending on seasonal conditions. Above 88% BOD removal efficiency was obtained at HRT = 3 h and 28°C from domestic wastewater, in a 20 L anaerobic biofilter using porous volcanic gravel [33]. The biofilter physically removes suspended solids, while biofilm is formed on the fibers and in the void matrix space. Using these same attributes, mineral wool has recently been studied as an aerobic biofilter matrix for municipal wastewater treatment [34,35]. At a constant surface flow rate of 0.07 m³/m²/min of domestic wastewater, a filter column of 2 m height and 0.38 m diameter was able to remove 43% of suspended solids, as well as 14%–25% of each of COD, BOD₅, NH₄-N, and PO₄-P, and

8%–13% of NO₃-N, depending on ambient temperature (that varied between 6°C and 16°C).

Biofiltration increases the proportion of stabilized organic matter, but the produced biomass itself is still unstable. A part of it may be recycled to the original anaerobic treatment, the rest needs post-treatment. The most appropriate post-treatment for later agricultural utilization is composting.

4.2. Composting

In the course of multiple bioconversion steps within the composting process, approximately 15% of the substrate organic content is ultimately built in stable organic macromolecules: humus precursors. The remaining organic content supplies energy for this bioconversion. The ratio depends on the technology applied, but in essence, it is due to aeration and temperature control.

Composting is analogous to natural soil formation. In natural soil ecosystems, organic matter is broken down by a number of processes: (a) building dissolved inorganic nutrients into living cells, (b) adsorption – eventually absorption – of suspended solids and colloids, (c) binding of extracellular organic molecules, and (d) decomposition of dead cell components take place simultaneously in the process of humification. Humus precursors are polycyclic, polyaromatic macromolecules with negative surface charge. Complexing with polyvalent metals, they can bind to each other, to other organic colloids and to silicate minerals to form aggregates of macroscopic size. The same takes place – *mutatis mutandis* – in the process of composting, which can therefore be regarded as a simplified, partial, but artificially accelerated soil formation.

INRA Narbonne (France) elaborated a remarkable methodology to understand aerobic stabilization of anaerobic effluents through the process of composting [12]. The carbon balance subset of compost characterization methodology was applied to study biogas digestate stabilization and agricultural applicability.

Industrial-scale composting of biodegradable organic materials has spread since the 1970s, becoming a mature aerobic stabilization process for a large variety of solid organic wastes. Also, compost is a manifest biofilter matrix material, since:

- it often contains the microbial strains necessary for the intended consortium; the selection pressure of the substrate submits those unnecessary for the treatment;
- its specific surface approaches that of soil, 80–500 m²/g,
- biofilm is easily formed on this surface;
- supplementary nutrients for biofilm formation are present in its material – in slowly released form.

The water retention capacity of compost is lower than that of mineral wool (up to 90%), it approaches only 60%. But its water conductivity (over 370 m/d), gas permeability (10⁻⁶–10⁻⁹ m²/Pa/s, depending on bulk density), and ion exchange capacity (CEC_{dm} > 70 cmol/kg) are all excellent [36]. This is why compost is a widespread matrix material in gas biofiltration. A more recent research path is biofiltration of liquids, such as domestic wastewater through use of a compost matrix [37,38]. Within a 6 weeks period of composting

municipal sewage sludge, a reduction in *Escherichia coli* > 2,400 MPN/g of sludge to approximately 20 MPN/g of compost material was obtained. TCOD removal of 10,450 mg/L was observed after a 60 d composting period, while the C/N ratio increased from 12.5 to 17.6.

4.3. Combination of biofiltration and composting

Combination of the above two methods facilitates simultaneous implementation of the four humus forming sub-processes. Composting biofiltration is expected to recover nutrients from the substrate by building the maximum possible quantity into stable organic macromolecule complexes. The COD – BOD₅ difference is expected to increase, indicating biological stabilization [12]. Nitrogen loss due to the narrow C/N ratio in the substrate is still unavoidable. Photosynthetic CO₂ binding, or some other additional carbon source would be necessary to eliminate such loss, these options are yet to be studied. Similarly, membrane technics could later be tested for CO₂ recycling, for microfiltration of the treated effluent, or to separate electrolytes in the case where composting biofiltration is combined with BES.

If agricultural utilization is the purpose, matrix material of the projected studies has a double task: both direct and indirect humus construction is to be verified. To this aim, mineral wool as an inorganic matrix and compost as an organic one stand out due to their unique properties of water retention capacity, water conductivity, air permeability, and ion exchange capacity.

5. Conclusions

With increasingly sophisticated technology, attested by more and more refined simulation methods [39], the development of the biogas sector has reached the level where insufficient utilization of digestates limit the further spread of the technology. With less than 400 biohydrogen installations compared to the nearly 20,000 biogas plants in the EU in 2015, biohydrogen industry is in the initial phase. It is, however, growing, and the problem of effluent treatment grows with. Due to their undeniable advantages, BES have been intensively researched for over a decade. Industrial-scale BES applications are expected to appear in the near future, and their effluents will unavoidably present the same difficulties.

These anaerobic effluents represent a high economic potential that justifies their consideration as secondary raw material, rather than pollutants. Their research needs to be refocused from “decontamination,” or “elimination,” to nutrient recovery.

Strengths and limitations of various nutrient recovery methods have been studied in this review. With the specific objective of agricultural utilization, microbiological stabilization methods – augmented biofiltration and composting – have been identified as the most promising for further tests given the current state of art.

Acknowledgments

This research is supported by project No. GINOP-2.3.2-15-2016-00016 “Water resource protection, development of

mobile water treatment systems and wastewater treatment technologies based on the University of Pannonia, in order to facilitate a dynamic growth of Hungarian export.”

Dr. István Molnár, honorary associate professor, director of “Agrár-Béta” Ltd. (Hungary) contributed with information on issues of industrial-scale anaerobic energy production that need further research.

Dipl. Eng. Jenő Farkas, retired head of the biotechnical research laboratory at the State Farm of Badacsony (Hungary) shared experience on agrochemical assessment of organic plant nutritives.

Dr. Nathalie Wéry, a research scientist at INRA Narbonne (France) shared her experience in compost characterization.

Dr. Tamás Rózsenszky, researcher at the University of Pannonia, communicated unpublished data on residual BOD content of microbial fuel cell effluents.

Dipl. Eng. Nathan Kunin, Leeds (UK), revised the paper for stylistic improvement.

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