

http://www.uem.br/acta ISSN printed: 1806-2563 ISSN on-line: 1807-8664 Doi: 10.4025/actascitechnol.v35i3.14069

Physicochemical study of pH, alkalinity and total acidity in a system composed of Anaerobic Baffled Reactor (ABR) in series with Upflow Anaerobic Sludge Blanket reactor (UASB) in the treatment of pig farming wastewater

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ABSTRACT. The anaerobic digestion efficiency varies according to several factors, such as: substrate carbon / nitrogen ratio, temperature, pH, alkalinity, and acidity. The main objective of this study was to describe the behavior of pH, alkalinity and total acidity of the affluent and effluent of a swine wastewater treatment system, in order to better understand the physicochemical process. The pH was measured immediately after collecting, and the methods of Jenkins et al. (1983) and Ripley et al. (1986) were used for quantifying the alkalinity, and the potentiometric method, for the acidity. The treatment system worked without large pH variation concerning the affluent and effluent of each unit, indicating good buffering conditions. The Ripley ratio (IA / PA) is characteristic for each effluent and dependent on the stage at which the reactor is working, and was determined as 1.96 for hydrolysis and acidification tank, 1.56 for reactors working on first stage (ABR), and 1.44 for reactors working on second stage (UASB).

Keywords: Ripley alkalinity, reactor buffering capacity, volatile fatty acids, bicarbonate alkalinity, wastewater treatment.

Estudo físico-químico do pH, alcalinidades e acidez total em sistema composto por Reator Anaeróbio Compartimentado (RAC) em série com Reator Anaeróbio de Manta de Lodo (UASB) no tratamento de águas residuárias de suinocultura

RESUMO. A eficiência do processo de digestão anaeróbia varia de acordo com uma série de fatores, tais como a relação carbono/nitrogênio do substrato, a temperatura, o pH, a alcalinidade e a acidez do meio. Neste sentido, este trabalho teve por objetivo estudar e descrever o comportamento do pH, da alcalinidade e da acidez total do afluente e do efluente de um sistema de tratamento de efluente de suinocultura, para melhor compreensão do processo físico-químico. O pH foi medido imediatamente após a coleta. Os métodos propostos por Jenkins et al. (1983) e Ripley et al. (1986) foram utilizados para quantificar a alcalinidade e o método potenciométrico para quantificar a acidez. O sistema de tratamento operou sem grandes variações de pH do afluente e do efluente de cada unidade, demonstrando boas condições de tamponamento. Observou-se que a relação de Ripley (AI/AP) é característica para cada efluente e varia em função do estágio em que o reator está operando, determinando neste trabalho relação AI/AP igual a 1,96 para tanques de hidrolisação e acidificação, 1,56 para reatores que operam em primeiro estágio (RAC) e 1,44 para reatores que operam em segundo estágio (UASB).

Palavras-chave: alcalinidade Ripley, tamponamento de reatores, ácidos graxos voláteis, alcalinidade bicarbonato, tratamento de efluente.

Introduction

Much attention has been given to swine feedlots by environmental control offices, owed the high pollution potential from this human activity. The current law requires the treatment of waste prior to discharge in water bodies, in order to prevent pronounced environmental problems (PEREIRA et al., 2009, 2010a, b and c, 2011). In this context, the anaerobic digestion appears as a great option, because significantly reduces the pollution potential, generates energy in the form of biogas, and allows the reuse of the treated effluent in fertigation. The biogas produced is frequently being used as a substitute of natural gas, especially in Europe (CAMPOS et al., 2010; SILVA et al., 2011a and b).

The anaerobic digestion is a biological process, occurring in the absence of oxygen, where

facultative or strictly anaerobic bacteria degrade complex organic compounds, converting them to biogas comprised of methane (60-7-%), carbon dioxide (40-30%) and other mineralized by-products (PEREIRA et al., 2010c).

The overall composition of the biogas produced by the ABR (Anaerobic Baffled Reactor) and by the UASB (Upflow Anaerobic Sludge Blanket reactor) varies according to several sources, such as: chemical composition of the effluent to be treated, substrate carbon/nitrogen ratio, temperature, pH, alkalinity, total acidity, operating parameters of the reactor and type of reactor, among others (PEREIRA et al., 2009a and b).

The alkalinity is the ability of a solution to neutralize acids, preventing pH variations given an increased concentration of acids (AQUINO; CHERNICHARO, 2005). Therefore, it is directly related to pH.

In monitoring the anaerobic reactors, the systematic appraisal of alkalinity becomes more important than the assessment of pH, since the pH values imply the consumption of high amount of alkalinity, reducing the buffering capacity of the medium (PEREIRA et al., 2009).

Bacteria producing methane have an optimal growth in the pH range 6.6-7.5, although the stabilization of methane production may be kept with pH between 6.0 and 8.0. pH values below 6.0 and above 8.3 should be avoided so that methanogenic bacteria are not inhibited (VON SPERLING, 2005).

In this way, this study examined and described the behavior of pH, alkalinity, and total acidity of a swine wastewater treatment system, in order to better understand the relationship between these parameters and the biological treatment process.

Material and methods

Pilot system for treatment of pig farming wastewater

The distance between the pig farming and the inflow of the treatment system had about 115 m. Initially, the pig farming wastewater (PFW) went through a preliminary treatment by a series of two units: a box to retain abrasive materials or Sand Box (SB) and a static screen (SS). The SB was 2.20 long and 0.53 wide. At 1.68 m from its entrance, there was a trough type Thompson made of slate, for measuring the flow rate, whose triangle mouth had 0.195 m in base and 0.095 in height.

Then, the PFW was conducted to SS, whose mesh was built in steel, 0.4 thick at the top, and 0.5 at the bottom. The steel rods that make up the mesh had a trapezoidal shape, with height of 2.5 mm,

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1.5 mm (largest basis) and 0.7 mm (smallest basis). These rods were welded into a steel bar with diameter of 3.8 x 17 mm, spaced at 3 cm. The support of the screen was constructed with PVC, 8 mm-plate, and the feed was given at the top, where there was a container 240 mm deep, where the influent wastewater was released and overflowed over the steel mesh in curved profile, where occurred the separation between liquid and solid portion.

The liquid portion was taken, through the holes of the screen, into the PVC box, whose structure supported the screen mesh and, through the bottom of this box, the PFW was taken to the acidification and equalization tank (AET) with volume of 8,000 liters.

After the effluent equalization into the AET, the PFW was pumped into the Anaerobic Baffled Reactor (ABR) using a Nemo pump (Netzsch, model NM015by01L06b), controlled by a frequency inverter (WEG-CFW08) with 12 inputs.

The ABR had three compartments, whose flow was upward and equaled through leveled gutters, constructed with several triangular weirs, aiming to homogenize the flow in each compartment to avoid dead zones and a hydraulic short-circuits (PEREIRA et al., 2010b).

This reactor was constructed with solid bricks, laid with cement mortar, internally coated with asphalt blanket. In order to avoid leakage and improve sealing, it was necessary to coat it with glass fiber. The first compartment of the ABR had 2,180 liters in volume; the second, 1,996 liters, corresponding to a total volume of 6,082 liters. The cross sections had the following dimensions: 0.56 x 1.14 m; 0.69 x 1.14 m; 0.73 x 1.14 m, with areas corresponding to 0.638, 0.787 and 0.832 m². The working volume of the compartments 1, 2 and 3 were 1.721, 2.12 and 2.24 m³, respectively.

The ABR effluent was sent by gravity to an Upflow Anaerobic Sludge Blanket reactor (UASB) with useful volume of 3,815 liters. This was constructed with brick masonry, laid with mortar, waterproofed with asphaltic blanket and then coated with fiber glass.

At the top of the reactor, it was installed a threephase separator, made by a half vibrated concrete pipe, semicircular shape, and laid reversed, through which the produced biogas was collected and conducted through PVC pipes with 12.5 mm diameter, to the pressure equalizer, then to flow meters, and finally burned in a flare system (PEREIRA et al., 2010a and 2011).

For sampling the sludge profile, five sampling sites (records in PVC) were installed on the side wall

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of the reactor, identical to those of ABR, evenly distributed along the height, with the last site located at 1.05 m from the ground level, and equidistant from each other 0.20 m. After treated in the UASB reactor, the effluent was removed by gravity to a PVC decanter (DE) with capacity of 3,000 L, where the PFW was retained to be reused.

Sampling, physicochemical analyses and frequency

The sampling of the influent and effluent of each unit was performed as follows: on the sampling day, four samples were taken by the morning at 8:00, 9:00, 10:00, 11:30 and four samples by the afternoon: 13:00, 14:00, 15:00 and 16:30, and each simple sample had 250 mL. After each collection, pН and temperature were instantaneously measured. By collecting the last sample, all were put together and homogenized, forming thus a single sample of 2 liters, which was used for all analyses. The preservation of the samples was done according to APHA (2005). Physico-chemical analyses, frequency, and methodology used are listed in Table 1.

Table 1. Parameters, frequency, and methods used in thephysico-chemical and hydraulic monitoring of the effluent duringthe study.

Physical and chemical parameters	Frequency	References
рН	Twice a week	APHA (2005)
Total (TA), partial (PA), and intermediate alkalinity (IA)	Twice a week	Ripley et al. (1986) and Jenkins et al. (1983)
Chemical Oxygen Demand (COD)	Twice a week	APHA (2005)
Biochemical Oxygen Demand (DBO ₅ ^{20°C})	weekly	Azide-modified Winkler method
Total acidity	Twice a week	APHA (2005), (potentiometric method with NaOH 0.02 N)
Flow	daily	Gravimetry

The operating parameters like Organic Loading Rate, Hydraulic Load (HL), and Hydraulic Retention Time (HRT) were calculated as established by Chernicharo (2007).

Results and discussion

Removal efficiencies of $BOD_5^{20^{\circ}C}$ and COD were 91.50 and 85.24%, respectively. The ABR and UASB reactors operated with HRT of 15.4 and 9.7 hours, respectively, HL of 1.57 m³ m⁻³ d⁻¹ for ABR and 2.5 m³ m⁻³ d⁻¹ for UASB; OLR of 4.46 kg m⁻³ day⁻¹ for ABR and 1.77 kg m⁻³ day⁻¹ for UASB.

The mean, maximal, and minimal room temperatures were 22.5, 29.5 and 17.3°C, respectively, and the relative humidity corresponded to 76, 97 and 34%. The ABR operated with sludge at average

The behavior of pH during the treatment is illustrated in Figure 1. The effluent reached the SB with pH between 7.5 and 8.5, in the alkaline range. This alkaline characteristic may have been caused by the use of detergents when washing the piggery, and by the food leftovers, which consist of minerals.

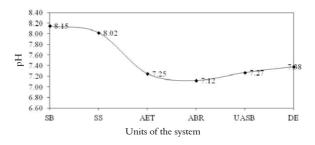


Figure 1. Variation of pH in the swine wastewater treatment system, during the monitoring (result relative to the mean of 16 data). Abbreviations according to the text.

On the static screen, the pH values remained between 7.4 and 8.3. A significant drop in pH was observed in the AET, being between 6.97 and 7.8, due to the formation of acetate, with consequent release of hydrogen. There was also the degradation of solids, increasing the nitrogen content. Since most of the total nitrogen was in the form of ammonia (NH₃), an increase in the concentration of N2 took place, due to the disruption of nitrogen-hydrogen bonds of the ammonia, forming thus H₂ and H₃O⁺, reducing the pH of this unit. After the AET, the effluent reached the ABR, which presented a better hydrolysis and acidification, and the pH remained between 6.86 and 7.5. In most of the monitoring, the ABR operated with pH of 7.1, within neutral range. Minimum and maximum values of pH were associated to imbalances in the system, caused by rainfall, breaks for washing, and unclogging pumps. As the ABR has great ability to absorb organic and hydraulic shocks, even with the UASB working in series, during the peaks and falls of pH in the ABR, no interference of the UASB was found, and the pH value remained between 6.93 and 7.5, that is, within the optimum range for growth of microorganism producers of methane (PEREIRA et al., 2009).

The DE, placed after the UASB, where occurred the final removal of solids, operated with pH between 7.00 and 7.87.

The anaerobic reactors (ABR and UASB) showed good buffering conditions, which according to Campos et al. (2010), is very important when considered the maintenance cost, because under

great pH variations, it is required the addition of chemicals for buffering.

Behavior of total (AT), partial (PA) and intermediate alkalinity (IA)

In the SB, the values of total, partial and intermediate alkalinity were the highest observed in the treatment system, reaching peaks of 6,453; 2,500 and 5,568 mg CaCO₃ L⁻¹ respectively. A drop in the values was verified when the effluent went through the static screen that remove the coarse solids, obtaining the maximum values of 3,874; 1,730 and 3,020 mg CaCO₃ L⁻¹ for total, partial and intermediate alkalinity.

At the AET a short and partial hydrolysis starts, along with a partial acidification of the nutrients. However, as the swine feed is rich in minerals like Na⁺, Ca²⁺, Mg²⁺ etc., after the partial hydrolysis is common the formation of intermediate volatile fatty acids, which when underwent biochemical conversion, make up compounds that generate alkalinity, such as sodium bicarbonate as shown in Equation 1 (CHERNICHARO, 2007).

$$CH_{3}COONa + H_{2}O \rightarrow CH_{4} + CO_{2} + + NaOH \rightarrow CH_{4} + NaHCO_{3}$$
(1)

Besides that, the breakdown of proteins, a nutrient common in swine feed, results in amino acids which with biochemical conversion make up ammonium (NH_4^+), also generate alkalinity owing the combination between ammonium and carbonic acid in solution, forming ammonium bicarbonate (Equation 2), justifying thus the behavior of concentrated alkalinity in the AET (Figure 2).

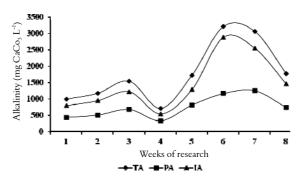
$$NH_3 + H_2O + CO_2 \rightarrow NH_4^+ + HCO_3^-$$
(2)

The degradation of carbohydrate and alcohol do not generate alkalinity by being organic compounds (CHERNICHARO, 2007; PEREIRA et al., 2009) without metal ions bound in their structures, thus they have only acid importance, when chemically analyzed the relationship of these compound with the sludge in the AET, due to the release of hydronium (H_3O^+) by the degradation.

As the tank is under constant supply, during the hydrolysis and acidification of nutrients, the effluent is equalized, generating a mixture and leakage of its fluid that was degraded in the AET with short HRT. This parameter could not be measured due to the hydrodynamic features of this unit.

Data presented as weekly results in Figure 2 refer to the average of two samplings made within one week. As already cited, the effluent treatment system receives the waste from a pig farming that operates

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acidity, being registered an increase in the values of

all parameters during these weeks.

Figure 2. Variation of total, partial, and intermediate alkalinity in the AET, at the beginning of biological treatment process. Abbreviations according to the text.

In the Figure 3 are shown the variation of total, partial and intermediate alkalinity of the ABR and UASB of the swine wastewater treatment system.

The ABR accounted for a more effective phase of hydrolysis and acidification, the products were metabolized by fermentative microorganisms, and converted into simpler compounds, like volatile acid, alcohol, lactic acid, carbon dioxide, hydrogen, ammonium, and hydrogen sulfide. These compounds reached the USBA and were used by the bacteria and methanogenic archaea to produce biogas, resulting in the alkalinities shown in the Figure 3.

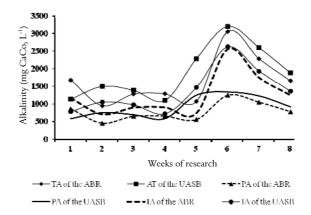


Figure 3. Variation of total, partial, and intermediate alkalinity of the ABR and UASB of the swine wastewater treatment system. Abbreviations according to the text.

After the treatment in the UASB, the main solids were its biomass removed by wash out and the effluent was submitted to a physical process of solids

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removal in the decanter, wich mean values, for the total, partial, and intermediate alkalinity were 1,693, 788 and 1,274 mg CaCO3 L^{-1} , respectively.

Ratio IA/PA in the treatment system

The alkalinity determination in two stages leads to the ratio IA/PA. According to Ripley et al. (1986) values of IA/PA above 0.3 for domestic sewage indicate disturbances in the anaerobic digestion. Nevertheless, Pereira et al. (2009) stated that it is possible to achieve a stability in the process with values different from 0.3, due to variations in the characteristics of each effluent.

By the start of the study, the AET kept the stability in the values of IA/PA, but, at the end, this stability was affected by the significant increase in volatile acids. This because in this period the sludge of the AET was more adapted to the effluent and the degradation of compounds was greater, pointing out a high concentration.

The ABR had always a ratio IA/PA lower than the AET, possibly owing the increased value of bicarbonate alkalinity (Figure 4). Due to the trend to balance of the values of IA and PA during the monitoring, it was verified a continuous decrease in the ratio IA/PA, according to the process of removing organic material.

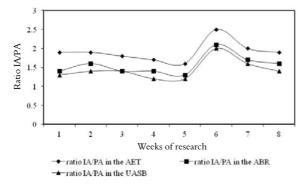


Figure 4. Variation of the ratio IA/PA in the units AET, ABR and during the study. Abbreviations according to the text.

Santana and Oliveira (2005), Fernandes and Oliveira (2006), and Lourenço and Campos (2009) found values for the ratio IA/PA in UASB reactors treating wastewater from pig farming of 0.47; 0.23 and 0.41 respectively.

In this study, the mean value recorded for the ratio IA/PA was 1.44 without problems of physical and chemical imbalances in the UASB reactor and in the system, related to their good buffering capacity.

Total acidity

The acidity of an effluent is its ability to react to pH changes produced by the bases, especially due to the

presence of free carbon dioxide (CHERNICHARO, 2007), volatile fatty acids and alcohols. It is mainly associated with the presence of free carbon dioxide, volatile fatty acids (VFA) and hydrogen sulfide, produced by the digestion.

The balance of the total volatile acids (TVA) is important when studying anaerobic reactors, because high concentrations of TVA may affect the biochemical process and eventually disturb the anaerobic digestion, which may lead the reactor to the collapse. The production of large amounts of VFA accelerates the activity of acetogenic bacteria, but inhibits methanogenic microorganisms, once they do not consume the acids resulting from acetogenesis in the same velocity they are produced (AQUINO; CHERNICHARO, 2005).

Biological treatment units belonging to the system AET, ABR and UASB have operated with total acidity values always below 100 mg L⁻¹ and the system worked well below the instability threshold, indicating an optimal performance. For Pereira et al. (2009) in the biological treatment systems, the accumulation of volatile acids above 150 mg L⁻¹ is the first evidence that the system is not working under ideal conditions.

The acidogenesis, an anaerobic digestion phase, started at the AET and continued in the ABR. In this phase, the VFA, alcohols, and mineral compounds are formed, as intermediate products, during the degradation of carbohydrate, protein, and lipid. These acids represent the compounds, from which most of the methane is produced by methanogenic bacteria in the UASB.

Organic and hydraulic overloads may cause imbalances in the process, which can result in the accumulation of volatile organic acids, especially propionic and butyric acids, and alcohols (LANGENHOFF et al., 2000). This was not observed in the ABR due to its ability to absorb organic and hydraulic loads, always working with low total acidity and optimal buffering ranges.

Methanogenic archaea, in sufficient amount and under suitable conditions, use intermediate acids as fast as they are formed, showing a good buffering capacity. A significant decrease was registered in the acidity of the ABR effluent when passing through the UASB, showing the consumption, especially of TVA, by methanogenic archaea, characterizing a stable condition in the reactor, even at the end of the experiment, when a peak in acidity did not exceed 80 mg L⁻¹ (Figure 5). In the Figure 5, each dot on the abscissa, relative to the study week, is equivalent to the average value among the data registered in that week.

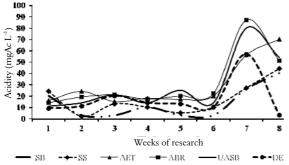


Figure 5. Variation of total acidity in the swine wastewater treatment system. Abbreviations according to the text.

Conclusion

The methodology proposed by Ripley et al. (1986) allowed more scientific knowledge on the alkalinity behavior through the pH ranges, in addition to showing a method more accurate than others that require color changes and are unfavorable to agroindustrial effluents, with high turbidity and color bands difficult to identify in situations of color change with methyl orange or phenolphthalein.

The use of ABR as pretreatment prevented alkalinity shocks and acidity, avoiding sudden variation in pH, which positively assisted the maintenance of microbial activity, especially methanogenic archaea, due to the system buffering. The buffering conditions had positive influence on biogas production. For the ratio IA/PA values of 1.96, 1.56 and 1.44 were found for the AET, ABR, and UASB, respectively, considered high values due to the composition of pig manure that varies according to ingested feed, and by the high organic load applied to the system.

The use of the AET as a mixed treatment (primary due to settling and secondary due to the formation of activated sludge through settled solids) assisted in the acidification and homogenization of the effluent, being responsible for the optimization of the secondary process.

Acknowledgements

The authors are grateful to the Laboratory of Water Analyses of the Engineering Department (LAADEG/UFLA) for performing the analyses, to the FAPEMIG for funding the facilities and equipment of the Pilot System for Treatment, through the Project TEC 1550/03. Thanks are extended to CNPq for granting scholarships.

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Received on July 19, 2011. Accepted on April 27, 2012.

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