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EFFECT OF BICARBONATE ALKALINITY ON GRAVIMETRIC SOLIDS ANALYSIS IN ANAEROBIC WASTEWATER TREATMENT

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SUMMARY

Bicarbonate alkalinity plays an important role in the stability of biological reactors used in wastewater treatment, primarily in anaerobic systems. As some wastewaters tend to acidify readily, addition of an external alkali source may be necessary to maintain process stability. An assessment was made of the effect of sodium bicarbonate addition on the determination of solids concentration. The methodology consisted in accompanying a series of solids concentrations (total solids, TS; total volatile solids, TVS; and total fixed solids, TFS) in samples containing cheese whey and volatile acids used to simulate anaerobic reactor effluents. TS, TVS and TFS showed to be strongly affected by $NaHCO_3$ addition, mainly due to an increase in TFS. This effect could be quantified by relating the experimental values to the theoretical ones from the stoichiometric equations for $NaHCO_3$ decomposition and other compounds (sodium acetate and sodium propionate) formation with temperature increase. In this way, as one of the main parameters of assessing liquid effluent treatment systems is the reduction in solids present in the medium, the concentration of solids can be quantified more adequately by determining fixed solids from the inorganic salts present. This methodology showed to be adequate in cases where a significant amount of alkali is added.

Introduction

The alkalinity of a liquid medium is related to its ability to neutralize acids and corresponds, mainly, to weak acid salts present in it. In anaerobic systems, bicarbonate alkalinity plays an important role, as it allows buffering of pH when volatile acids production becomes excessive.

The potential of a substrate to generate acids can be predicted by its composition. Carbohydrate-rich effluents have a great potential to acidify by fermentation in sewers and anaerobic treatment processes (Bagley and Brodkorb, 1999; Shizas and Bagley, 2002). As a result, in some situations, system stability is hard to maintain. In anaerobic reactors, when acids production exceeds acids consumption with significant reduction in pH, process breakdown may result. In these cases, it becomes necessary to either add external sources of alkalinity or to adopt adequate opera-

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EFECTO DE LA ALCALINIDAD BICARBONÁTICA SOBRE EL ANÁLISIS GRAVIMÉTRICO DE SÓLIDOS EN TRATAMIENTO ANAEROBIO DE AGUAS RESIDUALES

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RESUMEN

La alcalinidad bicarbonática tiene papel fundamental en la estabilidad de reactores biológicos aplicados al tratamiento de aguas residuales, principalmente en sistemas anaerobios. Como algunas aguas residuales pueden sufrir severa acidificación, en algunos casos es necesaria la adición de una fuente externa de alcalinidad para que el proceso sea conducido de forma estable. En ese contexto, se evaluó el efecto de la adición de bicarbonato de sodio sobre la determinación de la concentración de sólidos. La metodología consistió en la evaluación de las concentraciones de sólidos (sólidos totales - ST, sólidos volátiles totales - SVT y sólidos fijos totales - SFT) en muestras conteniendo suero de queso y ácidos volátiles (para simulación de efluentes de reactores anaerobios). Los valores de ST, SVT y SFT fueran fuertemente influenciados, principalmente debido al aumento de los SFT. Ese efecto fue cuantificado relacionándose los valores experimentales con los teóricos, determinados por las reacciones estequiométricas de la descomposición del bicarbonato de sodio y otros compuestos formados (acetato de sodio y propionato de sodio) con el aumento de la temperatura. Así, como uno de los principales parámetros de evaluación de sistemas de tratamiento de aguas residuales es la remoción de sólidos presentes en el medio, la concentración de sólidos puede ser evaluada de forma más adecuada teniendo en cuenta la determinación de los sólidos fijos debido a las sales inorgánicas. Esa metodología es considerada adecuada cuando se adiciona grande cantidad de alcalinizad a la agua residual.

EFEITO DA ALCALINIDADE À BICARBONATO SOBRE A ANÁLISE GRAVIMÉTRICA DE SÓLIDOS EM TRATAMENTO ANAERÓBIO DE ÁGUAS RESIDUÁRIAS

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RESUMO

A alcalinidade a bicarbonato exerce papel fundamental na estabilidade de reatores biológicos aplicados ao tratamento de águas residuárias, principalmente em sistemas anaeróbios. Como algumas águas residuárias possuem a tendência de acidificar rapidamente, em alguns casos faz-se necessária a adição de uma fonte externa de alcalinidade para que seja mantida a estabilidade do processo. Neste contexto, avaliou-se a influência da adição de bicarbonato de sódio na determinação da concentração de sólidos. A metodologia consistiu no acompanhamento das concentrações da série de sólidos (sólidos totais - ST, sólidos voláteis totais - SVT e sólidos fixos totais - SFT) em amostras contendo soro de queijo e ácidos voláteis (para simular efluentes de reatores anaeróbios). Os valores de ST, SVT e SFT foram fortemente influenciados, principalmente devido ao aumento dos SFT. Dessa forma, foi possível quantificar este efeito relacionando-se os valores experimentais com valores teóricos determinados a partir das equações estequiométricas de decomposição do bicarbonato de sódio e outros compostos formados (acetato de sódio e propionato de sódio) com o aumento da temperatura. Assim, como um dos principais parâmetros de avaliação de sistemas de tratamento de efluentes líquidos é a redução dos sólidos presentes no meio, torna-se possível quantificar de maneira mais adequada o seu valor pela determinação dos sólidos fixos devido aos sais inorgânicos presentes. Portanto, esta metodologia de análise mostrou-se adequada para as os casos em que a quantidade de alcalinizante adicionado é significativa.

tional strategies. (Ergüder *et al.*, 2000; Omil *et al.*, 2003; Demirel *et al.*, 2005).

Bicarbonate alkalinity supplementation can be done using several sources, namely: sodium bicarbonate, sodium carbonate, sodium hydroxide, magnesium oxide or lime. Despite the higher cost of sodium bicarbonate on an equivalent basis, its high solubility and lack of need for carbon dioxide neutralization make it easy to use with little risk (Speece, 1996).

The solids that are present in samples are an important environmental parameter since contaminants, with the exception of dissolved gases, contribute in one way or another to solid load (Metcalf and Eddy, 2003). Hence, the concentration of solids may be related to organic matter content, usually measured as chemical oxygen demand (COD). Van Haandel and Lettinga (1995) estimated a COD/volatile solids ratio of around 1.48, which is consistent with the COD of biomass. In a study on anaerobic treatment in ASBBR, Damasceno *et al.* (2007) observed that sodium bicarbonate addition to cheese whey caused a significant alteration in the experimental results for total solids, total volatile solids and total fixed solids, compromising the interpretation of the results, notably when related to their COD values.

In this context, the purpose of the present study was to assess the effect of sodium bicarbonate ($NaHCO_3$) addition on the concentrations of solids obtained by gravimetric analysis. Moreover, the feasibility of quantifying this effect using theoretical principles, as well as the experimental behavior of the existing salts in the samples, were explored.

Materials and Methods

The study comprised three parts, as follows:

Assay I

Assessment of $NaHCO_3$ in gravimetric analysis. The aim of this stage was to determine the behavior of $NaHCO_3$ when submitted to temperatures of 105 and 550°C, employed in the determination of the series of solids. Samples consisted of 200mg $NaHCO_3$ dissolved in distilled water.

Assay II

$NaHCO_3$ addition to organic substrate at different organic matter concentrations and supplementation ratios. The organic substrate consisted of reconstituted dehydrated cheese whey characterized by an equivalent organic load of 1mg-dry substrate per 1mg-COD (Ratusznei *et al.*, 2003; Damasceno *et al.*, 2007). The expected equivalent factor should be ~0.95mg-substrate per mg-COD, because the

TABLE I
MASS OF TOTAL SOLIDS (TS), TOTAL FIXED SOLIDS (TFS) AND TOTAL VOLATILE SOLIDS (TVS) DETERMINED IN ASSAY I*

Parameter	Mass (mg)
TS	123 ±1
TFS	120 ±1
TVS	3 ±2

* Number of samples= 12.

composition of whey contains carbohydrates (0.94mg per·mg-COD^{-1}) and casein (protein). As shown in Table II CODs of 4000, 6000 and 10000 presented 3718mg dry matter·per 4000mg COD (i.e., 0.93); 5698mg dry matter·per 6000mg COD (i.e., 0.95); and 9450mg dry matter·per 10000mg COD (i.e., 0.95). However, the value of 1mg dry substrate·per mg COD was maintained, due to the moisture of the whey of about 3-5% when the influent was prepared.

Bicarbonate supplementation was performed according to Ratusznei *et al.* (2003), in terms of $NaHCO_3$ mass to organic matter mass, measured as COD (mg HCO_3·per mg-COD). COD concentrations of 2000, 4000, 6000 and 10000mg·l^{-1} were assessed, with supplementation ratios of 0, 50 and 100%. For instance, at 2000mg COD per liter supplementation of 0% means that no $NaHCO_3$ was added, of 50% means that 1000mg $NaHCO_3$ was added and of 100% means 2000mg $NaHCO_3$ was added.

Assay III

$NaHCO_3$ addition to synthetic medium simulating anaerobic reactor effluent. The goal of this assay was to study the interaction of $NaHCO_3$ with other compounds that are eventually found in anaerobic wastewater treatment samples. Since the main acids formed during anaerobic fermentation of organic matter are acetic and propionic, the synthetic wastewater was prepared with these acids.

The medium consisted of 50wt% acetic acid and 50wt% propionic acid, at concentrations of 200, 400 and 1000mg·l^{-1}. The behavior of this medium was assessed with and without $NaHCO_3$. Supplementation was performed so as to result in an excess of $NaHCO_3$ corresponding to 50% of the added mass, according to the equations

$$CH_3COOH+NaHCO_3 \rightarrow CH_3COONa+H_2O+CO_2 \quad (1)$$

$$CH_3CH_2COOH+NaHCO_3 \rightarrow CH_3CH_2COONa+H_2O+CO_2 \quad (2)$$

According to Ec. 1 and 2, ~140 and 114mg $NaHCO_3$ would be necessary to completely neutralize 100mg of acetic and propionic acid, respectively. Neutralization involves permanence of the acid as salt, with no interference in the anaerobic process. Considering excess $NaHCO_3$ (50%), its concentration was calculated according to

$$[NaHCO_3]= 1.5\times(1.40g_{NaHCO_3}\times[HAc]+1.14g_{NaHCO_3}\times[HPr]) \quad (3)$$

The reaction of $NaHCO_3$ with acetic acid and propionic acid yields approximately 140mg sodium acetate and 130mg sodium propionate for each 100mg of acid, respectively, according to the stoichiometric Ec. 1 and 2.

To assess the behavior of the formed salts, the assay was carried out with 200mg sodium acetate and sodium propionate, in individual samples and without $NaHCO_3$ addition.

Analysis of total solids (TS), total fixed solids (TFS) and total volatile solids (TVS) were performed according to APHA (1995).

Results and Discussion

Table I lists the different solid masses determined from 200mg $NaHCO_3$ dissolved in distilled water. During determination of total solids (oven at 105°C), a loss of approximately 77 mg $NaHCO_3$ resulted, on average, corresponding to a reduction of 38.5%. According to Mackenzie (1970), when submitted to temperatures ~100°C sodium bicarbonate decomposes, generating sodium carbonate, carbon dioxide and water according to the following stoichiometric equation:

$$2NaHCO_3 \rightarrow Na_2CO_3+H_2O+CO_2 \quad (4)$$

Hence, for an initial mass of 200mg and Ec. 4, approximately 126mg would remain as Na_2CO_3. The experimental data confirmed the validity of this consideration, showing similar values (TS= 123 ±1). According to Budavari *et al.* (1996), Na_2CO_3 melts at 815°C, but at 400°C starts the loss of CO_2. This might explain the small variation in Na_2CO_3 mass (as TFS). The small loss, as TVS, would be related to release of CO_2.

Table II and Figure 1 show the concentrations of solids in reconstituted cheese whey samples at different organic matter concentrations and $NaHCO_3$ supplementations. The addition of $NaHCO_3$ significantly affected the concentrations of solids in cheese whey samples. The variation was mainly due to the increment observed in the total fixed solids (TFS). A similar behavior was observed with the different organic matter concentrations in the cheese whey samples, indicating that it was independent of the concentration of solids in the whey samples with no $NaHCO_3$ supplementation

The COD/volatile solids ratio, considering only the assays without supplementation, presented an average value of 1.1 ±0.05, showing that the ratio proposed by Van Haandel and Lettinga (1995) was not adequate in this case. Since these authors determined this value in samples containing biomass, the behavior was expected to be different.

Assuming that the added $NaHCO_3$ did not react with the whey constituents, alterations in analysis results would be proportional to the amount of bicarbonate added to the sample. Considering the results shown in Table

TABLE II
CONCENTRATIONS OF TS, TFS AND TVS IN CHEESE WHEY SAMPLES WITH DIFFERENT ORGANIC MATTER CONCENTRATIONS, AS COD, AND DIFFERENT $NaHCO_3$ SUPPLEMENTATION RATIOS

COD (mg·l^{-1})	$NaHCO_3$ (mg·l^{-1})	TS* (mg·l^{-1})	TFS* (mg·l^{-1})	TVS* (mg·l^{-1})
2000	0	2010 ± 33	130 ± 9	1880 ± 41
	1000	2222 ± 55	652 ± 53	1570 ±106
	2000	3050 ± 95	1283 ± 18	1767 ±105
4000	0	3718 ±113	262 ± 28	3457 ±141
	2000	4532 ± 60	1758 ±335	2773 ±354
	4000	5972 ± 76	2558 ±116	3413 ±192
6000	0	5697 ±123	473 ± 12	5223 ±112
	3000	6830 ±236	2223 ± 84	4607 ±160
	6000	8717 ±172	3960 ± 44	4757 ±207
10000	0	9450 ± 98	753 ± 58	8697 ± 45
	5000	10940 ±235	3740 ±192	7200 ± 92
	10000	14637 ±104	6587 ±155	8050 ±185

* Number of samples: 3.

I, for every 1000mg $NaHCO_3$ supplemented, approximately 615mg would remain as total solids, distributed as 600mg of total fixed solids and 15mg of total volatile solids. To assess this assumption, Figure 2 was elaborated showing the experimental values as a function of the theoretically expected values. The latter were calculated from the sum of the solids determined in the cheese whey with no supplementation and the values expected from Ec. 4, determined in assay I. The fit between experimental and theoretical values showed to be satisfactory, especially for the total fixed solids (TFS).

However, Table II also shows that the total volatile solids (TVS) is the parameter that is the least influenced by bicarbonate addition. Given that TVS may be used for approximate calculations of organic matter removal, it seems that TVS can still be utilized for this purpose, independently of bicarbonate levels. Another observation is that, against expectations, the addition of bicarbonate for a given COD leads to decreased values of TVS. The bicarbonate addition effect on TVS values should be very small, since according to Table I this would be related to CO_2 release. Hence, it is likely that there is some interaction between bicarbonate and the original organic matter, probably between the lactic acid present in the whey and bicarbonate, where the neutralization reaction leads to CO_2 formation with a further decrease in mass. This effect becomes less pronounced with increasing amounts of bicarbonate, as the amount of whey remains the same whereas the amount of bicarbonate is higher.

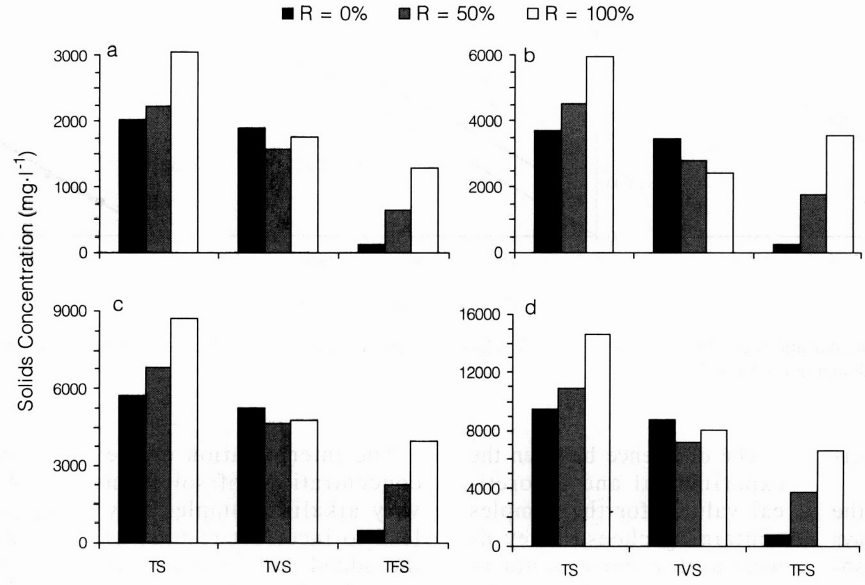

Figure 1. Effect of $NaHCO_3$ addition on total solids (TS), total fixed solids (TFS) and total volatile solids (TVS) for organic matter concentrations, in terms of COD, of a: 2000mg·l^{-1}, b: 4000mg·l^{-1}, c: 6000mg·l^{-1} and d: 10000mg·l^{-1}.

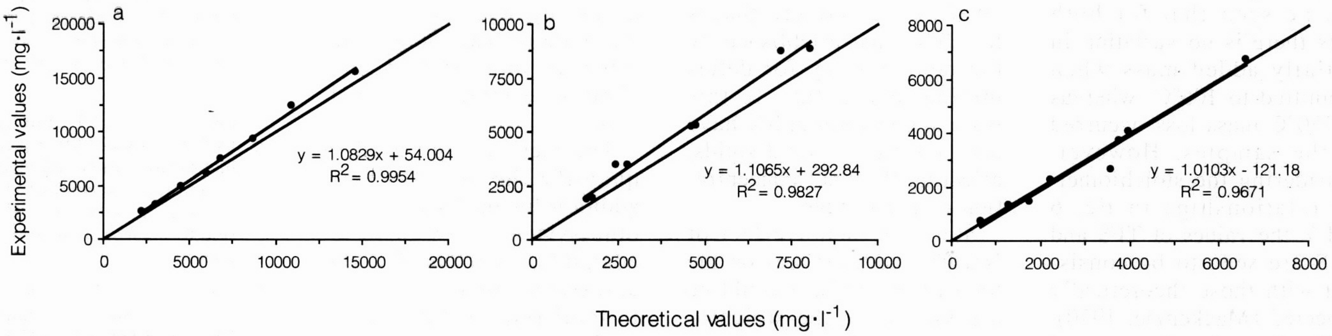

Figure 2. Dispersions of experimental and theoretical values of a: total solids, b: total volatile solids, and c: total fixed solids, in cheese whey samples supplemented with $NaHCO_3$.

TABLE III
CONCENTRATIONS OF TS, TFS AND TVS IN THE SUBSTRATE SIMULATING ANAEROBIC REACTOR EFFLUENT AT DIFFERENT TVA CONCENTRATIONS AND DIFFERENT $NaHCO_3$ SUPPLEMENTATION RATIOS

TVA (mg·l^{-1})*	$NaHCO_3$ (mg·l^{-1})**	TS*** (mg·l^{-1})	TFS*** (mg·l^{-1})	TVS*** (mg·l^{-1})
200	0	35 ±19	0	35 ± 19
	381	419 ±12	241 ± 3	178 ± 12
400	0	47 ±14	0	47 ± 14
	762	833 ±45	411 ±103	423 ±147
1000	0	32 ± 0	0	32 ± 0
	1905	1839 ±41	1197 ± 6	642 ± 46

* 50% acetic acid and 50% propionic acid.
** $[NaHCO_3] = 1.5 \times (1.40g_{NaHCO_3} \times [HAc] + 1.14g_{NaHCO_3} \times [HPr])$.
*** Number of samples= 3.

TABLE IV
CONCENTRATIONS OF TS, TFS AND TVS DETERMINED IN THE ASSAY WITH $NaHCO_3$

Salt	TS* (mg·l^{-1})	TFS* (mg·l^{-1})	TVS* (mg·l^{-1})
Sodium acetate ($NaC_2H_3O_2$)	200 ±0.8	126 ± 3	74 ± 4
Sodium propionate ($NaC_3H_5O_2$)	201 ±0.4	109 ±0.4	92 ±0.6

Table III lists the concentrations of total solids (TS), total fixed solids (TFS) and total volatile solids (TVS) for the synthetic medium simulating some characteristics of anaerobic reactor effluent (organic matter, acids and salts) at different concentrations of total volatile acids (TVA) and different $NaHCO_3$ supplementation ratios. It should be mentioned that TVS is not a reliable parameter for TVA estimation and, in fact, TVS leads to an underestimation of TVA in anaerobic effluents, since TVA are semi-volatile compounds that are prone to evaporate during the drying and incineration laboratory processes. Furthermore, when the COD of an effluent is composed of TVA, $NaHCO_3$ interferes

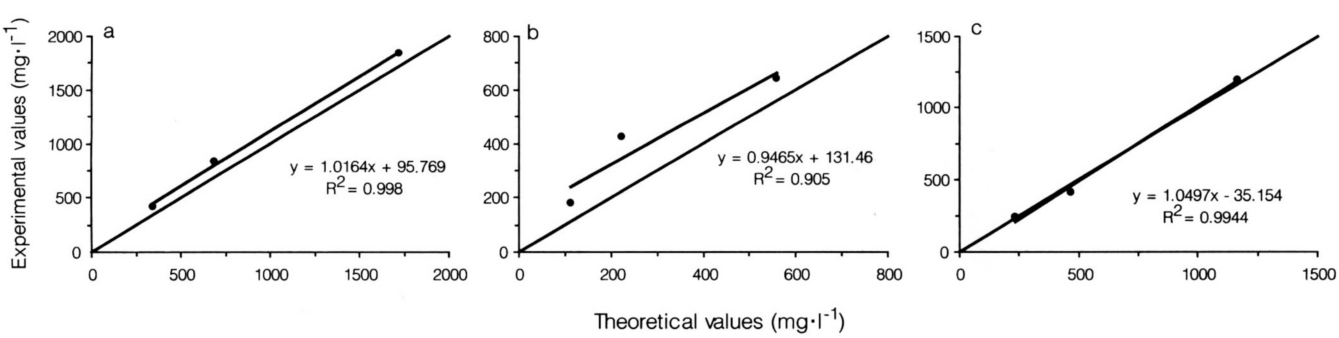

Figure 3. Dispersions of experimental and theoretical values of a: total solids, b: total volatile solids, and c: total fixed solids, in synthetic samples simulating anaerobic reactor effluents supplemented with $NaHCO_3$

when TVS is used for organic matter estimation.

In Table IV are shown the results obtained in the assays with sodium acetate and sodium propionate to evaluate the products formed during $NaHCO_3$ addition in samples containing volatile acids. It can be seen that for both salts there is no variation in initially added mass when submitted to 105°C, whereas at 550°C mass loss occurred in the samples. However, considering the stoichiometric relationships in Ec. 6 and 7, the values of TFS and TVS are seen to be consistent with those theoretically expected (Mackenzie, 1970), indicating that heating at 550°C resulted in the transformation of sodium acetate and sodium propionate into sodium carbonate.

$$2NaC_2H_3O_2 + 4O_2 \rightarrow Na_2CO_3 + 3H_2O + 3CO_2 \quad (6)$$

$$2NaC_3H_5O_2 + 7O_2 \rightarrow Na_2CO_3 + 5H_2O + 5CO_2 \quad (7)$$

Considering the behavior of the three salts, sodium bicarbonate, sodium acetate and sodium propionate, the concentrations of solids determined experimentally (data in Table III with supplementation) and those theoretically expected (from Ec. 4, 6 and 7) were plotted in dispersion graphs shown in Figure 3. Again, the fit showed to be satisfactory, especially for the total fixed solids data.

The difference between the experimental and theoretical values, for the samples containing cheese whey as well as for those containing volatile acids, can be explained by the fact that $NaHCO_3$ undergoes dehydration near 200°C (Mackenzie, 1970), a temperature that is far above that established by the methodology for determining total solids. In this way, water molecules have been counted as total solids, affecting the final concentration of the sample.

Since the major effect of $NaHCO_3$ addition was on the total fixed solids, it could be assessed from the stoichiometric equations as a function of temperature increase during gravimetric analysis, evidenced by a satisfactory fit between the experimental and theoretical values. As reduction of solids is an important monitoring parameter in liquid effluent treatment, it was possible to quantify the solids with higher accuracy.

Conclusions

Addition of sodium bicarbonate as an external source of alkalinity in cheese whey samples simulating samples rich in organic matter, contributed significantly to the determination of the concentrations of solids, mainly due to increase in total fixed solids. In effluents containing volatile acids, the concentration of solids was dependent upon the salts present as bicarbonate, acetate and propionate.

The interpretation of the concentrations of solids in very alkaline samples may lead to incorrect results, as the added and formed salts strongly affect the final result. Hence, utilization of this parameter as an operational tool in monitoring and assessing anaerobic reactors requires caution and the effect of interfering agents should be studied in each case.

The methodology used to quantify the effect of salt addition for buffering anaerobic systems showed to be adequate, as it enabled the assessment of the increase in total fixed solids during analysis and allowed a more correct determination of solids content.

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