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Article

Underestimation of Dry Matter of Anaerobic Media with High Bicarbonate Concentration

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Abstract: In the anaerobic digestion world, the dry matter (DM) and organic matter (OM) contents of the samples are major data for the design, handling, and maintenance of the processing of biogas plants. Volatile compounds, such as volatile fatty acids and ammonia, are likely to volatilize during drying at 105 °C and, thus, distort DM values; they have been described at length in the literature. On the other hand, the partial decomposition of bicarbonate, present in the digestion media, has been little described in the scientific literature. In this work, it appears that, in accordance with the stoichiometry of the partial decomposition of bicarbonate into CO₂ and H₂O, about 37% is volatilized as early as 70 °C and *a fortiori* at 105 °C, whether in aqueous solutions of pure bicarbonate or in different real digester media. With freeze-drying, no degradation of bicarbonate was observed, confirming the thermo-dependence of this reaction. This decomposition leads to an underestimation of DM measurements and some erroneous DM mass balances. It also led to an indirect overestimation of the OM, when expressed as a percentage of the DM.

Keywords: anaerobic digestion; volatile fatty acids; alkalinity; total solids



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1. Introduction

Anaerobic digestion (AD) is one promising biogas-producing process that performs wastewater or solid waste treatment. The characterization of the feedstock and digestate in anaerobic digesters is the key to a successful realization of AD. In the anaerobic digestion world, the dry matter (DM) and organic matter (OM) contents of anaerobic samples are the major data for the design, handling, and maintenance of the processing of biogas plants [1]. Many national and international standards and technical guidelines emphasize the importance of DM and OM measurement for the characterization of substrates, such as French norm NF EN 15934 [2], German standard VDI 4630 [3], and ISO 20675:2018 [4]. The determination of DM and OM in biowaste, such as sewage sludge, wastewater, animal slurries, and digestate, in wastewater treatment and biogas plants is usually achieved according to standard protocols [2,5]. Dry matter content (also put as total solids) is, therefore, determined by drying the samples at 105 °C until constant weight, usually expressed in the percentage of fresh weight. Organic matter content (also put as Volatile solids) is usually determined by burning dried samples in an oven at 550 °C until constant weight, expressed in the percentage of dry matter. Both DM and OM contents are used to realize the mass balance check in anaerobic digesters [6]. In addition, the biochemical methane potential (BMP) of one specific substrate is always reported and expressed in the

volume of methane produced by one gram, kilogram, or tonne of DM or OM [7–11]. They are among the first and most important parameters to be analyzed in biogas reactors at the industrial scale [12,13]. Hence, an inaccurate measure of DM or OM could inevitably lead to the wrongful interpretation of the experimental results and, therefore, a potential failure of anaerobic digestion.

However, these protocols were developed for sludge, biowaste, and other substrates containing relatively few substances that could interfere with the measurement. For instance, the French NF EN 15934 standard, related to “Sludge, treated biowaste, soil and waste—Calculation of dry matter content by determination of dry residue—or water content”, states that: “Samples may change during drying, e.g., by absorption of carbon dioxide in the case of alkaline samples or oxygen by reducing substances. Volatile compounds evaporating at 105 °C are measured as water according to the present procedure.” Obviously, loss of volatile compounds during DM determination may lead to important errors in mass balances. However, the standard protocols, as mentioned above, do not specify any potential volatile compounds influencing DM determination. The literature focused on ammonia, volatile acids, and alcohols. The proteinaceous biomass, made up of proteins such as shellfish sub-products and manures, will generate ammonium ions, which can be transformed into volatile ammonia. Considering the acidity constant of ammonium to ammonia ($pK_a = 9.24$ at 25 °C), the latter can be considered insignificant for pH below 8.0. However, as early as 1994, Derikx et al. [14] showed that all ammonia is lost for pH above 8.0 for pig, cattle, and poultry manure.

Volatile acids, whose pK_a are close to 4.8, can also affect the dry matter measurement. However, considering that volatility only concerns non-ionised fraction, a neutral pH would prevent their interference in the dry matter measurements.

Finally, the degradation of organics will generate carbonate and bicarbonate ions, which can be transformed into carbon dioxide. For samples highly charged of bicarbonate content, the thermal degradation of bicarbonate, during drying at 105 °C, could not be neglected. To our knowledge, no publication in the literature was interested in the possible interference of bicarbonate ions with DM measurements, which is the main topic of this work. The possible interferences of volatile acids were investigated, as well in the present paper.

Moreover, some authors recommend limiting the temperature to 80 °C and extending the drying time to avoid the loss of volatile compounds in silage and anaerobic media [15–19]. These papers are old and related to silage with high acid contents and low pH; therefore, they are somewhat marginal to our concerns. However, we decided to include the temperature factor in our work by conducting the tests at 70 and 105 °C.

The present work deals with the possible interference of bicarbonate and volatile acids in the determination of dry matter content in artificial aqueous solutions and real anaerobic samples. The results obtained in thermal drying were compared to those achieved by freeze-drying. The importance of taking into account the bicarbonate decomposition in calculating DM content has been underlined by a mass balance study in real AD media.

2. Materials and Methods

2.1. Preparation of Sodium Bicarbonate and Acetic Acid Solution

To study the influence of bicarbonate and acetic acid, sodium acetate trihydrate (ASC, ISO, Reag. Ph. Eur., Merck, Darmstadt, Germany), sodium bicarbonate (99.5%, Sigma, St. Louis, MO, USA), and deionized water at 18 M Ω ·cm were used to prepare the different solutions.

2.2. Volatile Fatty Acids Interference

As the pK_a of the VFAs are close, and so is their volatility, we only considered acetic acid in this work.

A 30 g/L acetic acid solution (i.e., 28.63 mL of 99.8% glacial acetic acid, i.e., about 0.5 mol/L) was made up; about 30 g of this solution was placed in crucibles and then

introduced into the oven (Memmert U10, Schwabag, Germany) at 70 and 105 °C overnight; the pH of the solution was 2.53. Although the NF EN 15934 standard states the drying must achieve up to constant weight, we previously determined that a 12-h drying (practically an overnight drying) would be sufficient for our samples (data not shown).

Similarly, about 30 g (30 mL) of an aqueous solution of 30 g/L sodium acetate was placed in crucibles and dried overnight at 70 and 105 °C. The pH of the solution was 9.23.

The analyses were performed in triplicate.

2.3. Bicarbonate Interference

Different solutions of sodium bicarbonate were made (10 g/L, 50 g/L), and aliquots were placed in crucibles before drying overnight at 70 and 105 °C. The 50 g/L solution was also freeze-dried (freeze-dryer Christ Alpha 2-4 LD Plus, Osterode am Harz, Germany), in order to compare dehydration methods. Finally, the freeze-dried residue was then placed overnight in an oven at 105 °C for comparison.

The decomposition of sodium bicarbonate was also evaluated, with a TGA-DTA SETSYS-1750 CS (Setaram Instrumentation, Caluire, France) and moisture analyzer (BEL Engineering Mark 160 Top Ray (Monza, Italy)).

As an example of biomass with high calcium carbonate content, we used blue mussels and slipper shells (from the company Cultimer Producteurs Associés, France) to verify the hypothesis of the dissolution of part of these shells with temperature and possible interference with the DM measurement.

To validate the conclusion drawn out with pure chemical, we used the effluent of the mesophilic pilot reactor treating shellfish substrates (data not shown). The effluent was freeze-dried and compared to the same effluent dried overnight at 105 °C. For both drying conditions, about 0.13 g of sodium bicarbonate had been added to about 10 g of effluent.

The analyses were carried out at least in triplicate.

3. Results and Discussion

3.1. Volatile Fatty Acids Interference

As mentioned in the introduction section, many authors have mentioned the loss of volatile fatty acids in their non-ionised form with temperature. However, most of the tests were carried out under standard drying conditions, i.e., at 105 °C; what about at a lower temperature? Additionally, it is often customary to put a lid on crucibles to avoid splashing of material with evaporation; would this be sufficient to limit the loss of VFA? Rather than testing different pH conditions, as Derikx et al. [14] did, only two conditions of pH and VFA form were tested, with pHs 2.53 and 9.23. Table 1 shows the mass losses for drying under the different conditions tested.

Table 1. Acetic acid and sodium acetate losses at 70 and 105 °C.

	Loss at 70 °C	Loss at 105 °C
Acetic acid solution (pH 2.53)		
crucible with lid	99.6 ± 0.1%	99.2 ± 0.2%
crucible without lid	99.9 ± 0.1%	99.5 ± 0.1%
Sodium acetate solution (pH 9.23)		
crucible with lid	1.55 ± 0.05%	1.35 ± 0.05%
crucible without lid	1.05 ± 0.05%	1.3 ± 0.1%

The results clearly show that acetic acid is completely lost from 70 °C onwards, whereas sodium acetate is only lost to a very small extent. On the other hand, the laboratory practice of placing a lid on the crucible, to prevent loss of material by boiling of the water in the solutions, does not modify the conclusions.

It could be explained by the fact that volatile fatty acids, in their ionised form, i.e., for solutions with pH values above about 7, do not significantly interfere with the DM

determination method. This confirms the previous findings, of Derikx et al. (1994) [14], regarding the loss of VFA during drying at various initial pH levels.

3.2. Bicarbonate Interference

3.2.1. Pure Bicarbonate Solutions

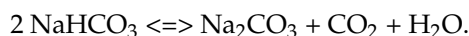
Sodium bicarbonate solutions at 10 g/L, i.e., concentrations in the order of magnitude of those found in anaerobic digesters, were in turn heated to 70 and 105 °C overnight. Table 2 shows the losses observed at 70 and 105 °C for the 10 g/L solutions, as well as for 2–5 g of mussel and slipper shells deposited in about 30 g of water.

Table 2. Bicarbonate and shell losses after drying overnight at 70 and 105 °C.

Samples Tested	Loss at 70 °C	Loss at 105 °C
Sodium bicarbonate solution (10 g/L)	35.9 ± 0.5%	35.9 ± 0.1%
Mussel shell	0.9 ± 0.1%	0.8 ± 0.3%
Slipper shell	0.7 ± 0.05%	0.7 ± 0.3%

These results show that bicarbonate decomposes, even at 70 °C, at a ratio of about 36%. This implies that the DM contents of a medium containing bicarbonate ions are correspondingly underestimated, especially for samples highly charged of bicarbonate content. To our knowledge, these surprising results have never been shown in the field of anaerobic digestion for biogas production.

In fact, the thermal decomposition of bicarbonate is well known and corresponds to the following stoichiometry:



This decomposition starts at 50 °C and increases from 70 °C [20,21]. It is at the origin of the outgassing of CO₂, for example, during the baking of bread.

Numerically, two moles of NaHCO₃, i.e., 168 g, will generate a loss of one mole of water and one mole of CO₂, i.e., a total of 62 g, or 36.9% of the initial mass. In anaerobic digestion media, bicarbonate is not systematically associated with the sodium ion; therefore, the loss of mass may be different, depending on the cations, but it will be of the same order of magnitude. For instance, the same calculation for potassium bicarbonate gives a mass loss of 31.0% (62 g loss as water and carbon dioxide for two moles of KHCO₃, i.e., 200 g).

Sodium carbonate is stable and will be found in the ash. This reaction result is, therefore, an underestimation of the dry matter content of media containing a lot of bicarbonate.

Finally, the results in Table 2 show that mussel or slipper shells do not interfere with the DM measurement. Solid calcium carbonate (the main constituent of the shell) is not affected by drying. As the decomposition reaction is identical at 70 and 105 °C, only the latter temperature was used for the next tests.

These tests were carried out with concentrations of 10 g/L, what about more concentrated solutions and, moreover, how to be sure that the mechanism is temperature-dependent? To answer those questions, new tests were conducted with an aliquot of a 50 g/L bicarbonate solution, heated to 105 °C overnight and freeze-dried in parallel. The freeze-dried bicarbonate solution was then heated to 105 °C overnight (Table 3).

Table 3. Bicarbonate losses of a concentrated solution after heat-drying and lyophilization.

Samples Tested	Loss at 105 °C	Loss after Freeze-Drying
Bicarbonate solution (50 g/L)	37.4 ± 0.0%	3.9 ± 0.3%
Bicarbonate (50 g/L) solution after freeze-drying	35.0 ± 0.3%	–

The results, using a five-more concentrated pure sodium bicarbonate solution, are identical to the previous ones, with an estimated loss of 37.4%. Conversely, freeze-dried

aliquots of this solution exhibited a loss of about 4%, confirming the hypothesis of a purely thermal decomposition of the bicarbonate, concomitant with the dehydration of the sample. The loss of 4% during freeze-drying is probably due to an experimental error, linked to the powdery side of the freeze-dried solution and, therefore, to weighing difficulties.

When the dry freeze-dried solid is later heated to 105 °C, under standard conditions, 35% of the bicarbonate decomposes, further confirming the thermo-dependent nature of the bicarbonate decomposition.

These tests were conducted with pure bicarbonate solutions in water, so four questions arise: firstly, whether this decomposition is observed in real anaerobic digestion effluents; secondly, to what extent is this decomposition of the bicarbonate and, therefore, this underestimation of DM detrimental; thirdly, how does this underestimation affect the measurement of OM; and, fourthly, at which temperature the decomposition begin?

3.2.2. Bicarbonate Thermal Losses in Real AD Media

To answer the first question, solid sodium bicarbonate was added to one aliquot of the effluent from two different methanogenic digesters, operating in stable mesophilic regimes. The first one treats blue mussels' sub-products, with a residence time of 20 days (data not shown); the second treats intermediate energy crops, with a residence time of around 40 days (data not shown). Solid sodium bicarbonate was added to aliquots of effluent, and the experimental DM contents of the effluents were subtracted from that of the sample with NaHCO₃ addition. The additional mass loss was attributed to the decomposition of the added bicarbonate.

The results of Table 4 confirm the results obtained with pure bicarbonate solutions; the differences between the losses can be attributed the precision of the weights, considering that for an effluent that contains 2% DM, the bicarbonate loss is about 0.05 g.

Table 4. Bicarbonate losses of a concentrated solution after heat-drying and lyophilization.

Samples Tested	Initial Fresh Mass (g)	DM (g)	Loss at 105 °C
	Effluent from the reactor treating blue mussel subproducts		
Sample 1	10.01 ± 0.01	0.21 ± 0.01	–
Sample 1 plus bicarbonate	12.55 ± 0.10	0.29 ± 0.01	37 ± 1%
Sample 2	50.00 ± 0.01	0.95 ± 0.01	–
Sample 2 plus bicarbonate	51.00 ± 0.01	1.63 ± 0.01	32 ± 1%
	Effluent from the reactor treating intermediate energy crops		
Effluent	50.00 ± 0.01	2.39 ± 0.01	–
Effluent plus bicarbonate	52.00 ± 0.01	3.59 ± 0.01	39 ± 1%

3.2.3. Importance and Effect of the Correction for Accurate DM Estimations

The question that arises is the interest and importance of the correction linked to the decomposition of the bicarbonate; is it significant, in practice, within the margin of error of the technique? To do so, the actual bicarbonate content of the medium must be first determined by an accurate method.

Bicarbonate can be determined by CIT, but titrimetric methods are the most used in anaerobic digestion, among others, the FOS/TAC method [22]. However, this method overestimates the bicarbonate values in a wide range, as shown by Liu et al. (2021) [23]. The calculation formulas developed by Liu et al. were applied to the influent and effluent of a 1000 L pilot digester, operating under steady state mesophilic conditions (data not shown), considering a uniform thermal decomposition of 37% of the sodium bicarbonate, measured by the modified FOS/TAC method. The measurements and balances, presented in Table 5, were obtained for a full-residence time (20 days).

Table 5. Mean bicarbonate content and overall DM and OM balances for a mesophilic pilot plant, considering or not the bicarbonate loss.

	[NaHCO ₃] (g/L)	Without Correction	With Correction
Influent, over 20 days feeding	average: 9.38	48.8 kg _{DM}	54.0 kg _{DM}
Effluent, over 20 days feeding	average: 18.2	38.4 kg _{DM}	48.8 kg _{DM}
DM conversion	–	21.4%	10.5%
OM conversion	–	21.7%	21.7%

The results show that when the bicarbonate content is important, these corrections are far from negligible; the DM contents are underestimated and the balances incorrect. The OM conversions are not affected, as discussed below.

3.2.4. Impact of Thermal Bicarbonate Decomposition on OM Values

The organic matter content (OM) is not intrinsically affected by bicarbonate decomposition, all the organic materials were burned between 105 and 550 °C [2,5], and as carbonates are stable at 550 °C, the decomposition of bicarbonate below 100 °C does not directly impact the OM determination. However, OM content is usually expressed as a percentage of DM and therefore it is affected through a ripple effect.

The Figure 1 below illustrates this paradox. Considering an anaerobic medium that contains 120 g DM, 72 g OM, and 10 g bicarbonate per liter. Weighing 10 mL in a crucible, i.e., 10 g (assuming the density of 1 g/mL), 1.2 g DM should remain in the crucible after drying at 105 °C, and 0.48 g OM should be weighted after burning at 550 °C, as shown in the blue boxes of Figure 1. Finally, the organic matter represents 7.2% of fresh matter (FM) and 60% of dry matter (DM). Considering now that 37% of the bicarbonate is lost during the drying (i.e., 0.037 g), as shown in the green boxes in Figure 1, for the same amount of 10 g in the crucible, 1.163 g DM will remain in the crucible after drying at 105 °C, and 0.44 g OM will be weighted after burning at 550 °C, giving an OM content of 7.2% FM and 61.9% DM.

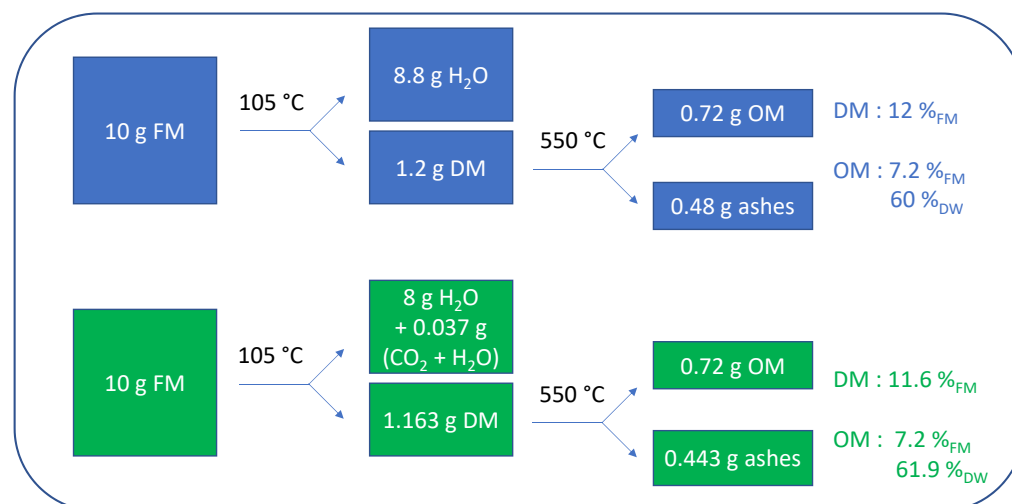


Figure 1. Schematic DM and OM balances, considering the bicarbonate decomposition.

The balances corresponding to the green boxes show that the experimental DM content is underestimated, and the OM content, expressed as % of DM, is inversely overestimated.

3.2.5. Influence of Temperature on the Bicarbonate Decomposition

Finally, one question that remains concerns the temperature at which this decomposition takes place. It has been shown above that the loss of bicarbonate is significant as early as 75 °C. This concerns the measurement of dry matter; however, if this decomposition were

significant, for example at 55 °C, systematic losses of buffering capacity could be observed in thermophilic reactors. To clarify this point, two complementary techniques were implemented: firstly, a measurement of the mass loss of aqueous solutions of sodium bicarbonate and solid sodium bicarbonate for different temperatures, using a thermo-balance; and, secondly, a measurement of the mass loss of a sample of solid sodium bicarbonate with a TGA/DTA device.

Approximately 4 g of solid sodium bicarbonate was placed in the moisture analyzer cup and heated to 50, 60, and 70 °C for 1 h. Figure 2a shows that no mass loss was observed at 50 and 60 °C and 10 ± 1% was lost at 70 °C after about one hour. This result is not contradictory to the results of paragraph 3.2.1, in which the solid bicarbonate samples were placed in the oven overnight. These results suggest slow kinetics, which will be checked using TGA/DTA, as discussed below. These experiments were performed in triplicate.

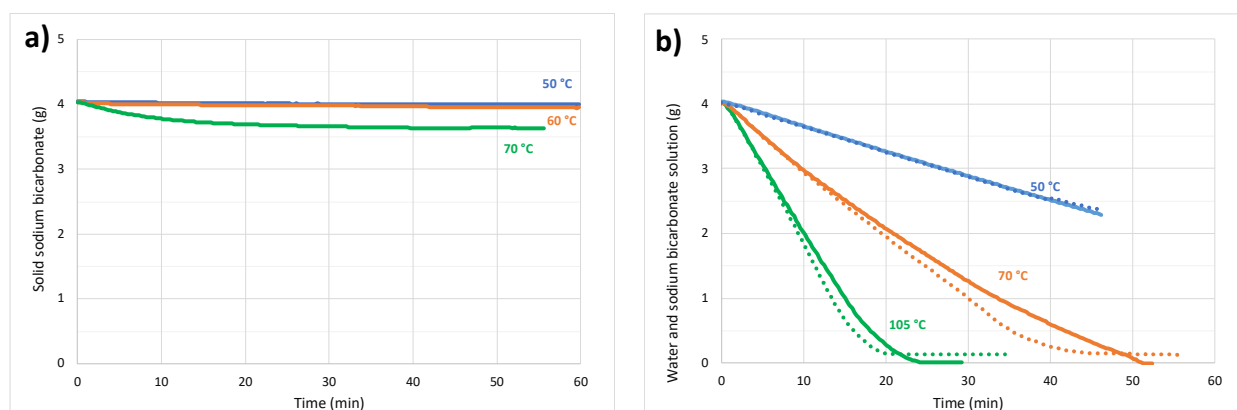


Figure 2. (a) Mass evolution of 4 g of solid sodium bicarbonate in the moisture analyzer, set at 50 °C (blue line), 60 °C (orange line), and 70 °C (green line). (b) Mass evolution of 4 g of distilled water (continuous lines) and 4 g of 5% sodium bicarbonate solution (dotted lines) in the moisture analyzer, set at 50 °C (blue), 70 °C (orange), and 105 °C (green).

Figure 2b shows that at 50 °C in 40 min, the mass loss is statically negligible, but it is rapid at 70 and 105 °C. After about 40 min at 70 °C and 22 min at 105 °C, the masses of the solutions stabilise at about 0.13 ± 0.01 g, which means a loss of bicarbonate of 0.07 ± 0.01 g or $35 \pm 5\%$. The precision of the instrument does not allow for a better accuracy. For technical reasons, it was unfortunately not possible to test other temperatures between 50 and 70 °C.

To confirm the temperature at which the decomposition reaction starts significantly, a 117 mg sample of solid sodium bicarbonate was placed in the TGA/DTA cup. Given the maximum mass allowed by the apparatus, a solution could not have been used, as the loss of mass due to decomposition would not have been measurable. The apparatus was programmed to stabilise for one hour at 50 °C, rise to 55 °C in 5 min, stabilise again for one hour at 55 °C, and so on, in successive steps of 5 °C up to 105 °C, finally maintained for 2 h (Figure 3).

The figure shows that the mass does not vary up to a temperature of 70–75 °C and kinetics are relatively slow; the loss of mass is 32% at 105 °C after 14 h of this experiment.

These results show that sodium bicarbonate decomposition only manifests itself measurably at temperatures above 70 °C, meaning that this loss will influence DM measurements by the proper functioning of digesters operating under thermophilic conditions at 55 °C.

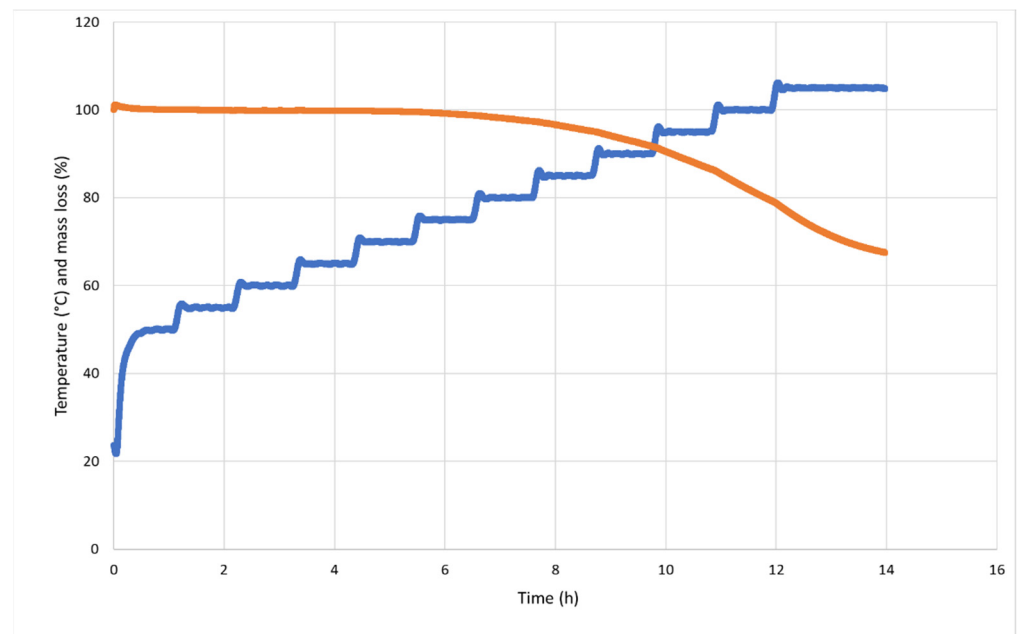


Figure 3. Temperature (blue line) and mass loss (orange line) of 117 mg of solid sodium bicarbonate in the TGA/DTA device.

4. Conclusions

The decomposition of bicarbonate is a uniquely thermo-dependent process that manifests itself measurably in dry mass measurements as early as 70 °C, let alone 105 °C. The resulting loss of mass during measurement is of the order of 35%, thus leading to an underestimation of the DM and indirect overestimation of the OM measurement in methanogenic reactors. For an anaerobic sample highly loaded with bicarbonate content, the drying at 105 °C, applying standard protocol, could give rise to a significantly wrongful results of DM and OM contents. This constitutes a major factor that should be considered when realizing mass balance in anaerobic reactors or reporting the specific methane potential of the sample concerned. It is also confirmed that no mass loss due to bicarbonate decomposition takes place at thermophilic conditions of anaerobic digestion.

These tests show that it makes sense to correct the experimentally obtained DM values, obtained with the standard method, by considering the bicarbonate content, since the other ions do not interfere with the measurement.

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