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Thermogravimetry as a technique for establishing the stabilization progress of sludge from wastewater treatment plants

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Abstract

Stabilization processes are key to the reliable performance of any wastewater treatment plant. However, a simple, yet reliable and widely acceptable analytical tool to evaluate the degree of stability reached during sewage sludge treatment remains to be found. Mesophilic aerobic digestion has been carried out on laboratory scale in aerated reactors using non-stabilized sludges from three different wastewater treatment plants. Temperature programmed combustion tests were carried out to investigate the stabilization degree of samples throughout the lab treatment. Differential thermal analysis (DTA) has shown that energy release during sludge combustion shifts to higher temperatures as stabilization advances. The integration of the DTA curve gives an area which is proportional to the energy released so it has been possible to quantify this shift by representing the percentage of the cumulative area at each temperature along each sample combustion. Although the initial composition of the sludge should be considered in each particular case, this preliminary study shows that thermogravimetry may offer a means of ascertaining whether an acceptable degree of stability has been reached by aerobic digestion.

Keywords: Sewage sludge; Stabilization; Aerobic digestion; Differential thermogravimetry; Differential thermal analysis

1. Introduction

Wastewater treatment is a solution to the pollution problems that would be brought about by the direct discharge of them onto shores and into rivers. As a by-product of treatment, sludges arise which have to be managed in a rational way in order not just to shift the problem from one area of the environment to another [1]. The final destination of these sludges will have to depend on their nature, whence the importance of characterizing them. In any event, the management principles applicable to any residue are applicable to sludge from water treatment, so, whenever possible, it should be made useful. The key for a successful use of biosolids is the production of a material of good quality, which depend on several parameters among which heavy metals content and stability are especially important.

The use of sewage sludge as organic-mineral fertilizer in the soil implies a usefulness dependent on that of the organic matter and nutrients contained in them, as well as the elimination of residues and a minimal environmental impact [2]. Different countries have different criteria for the agricultural use of biosolids (in the European Union, Council Directive 86/278/EEC of the 12 June 1986), whereby biological, chemical or thermal treatment is compulsory, as is long-term storage or any suitable process significantly

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reducing the fermentation power of sludge and the health risks inherent in its utilization. Such use therefore requires stabilization [3].

Stabilization is one of the stages in the treatment of sludge from wastewater treatment plants [4], the three aims of which are the reduction of pathogens, the elimination of unpleasant smells and the reduction or elimination of the potential for putrefaction.

The degree of stabilization achieved may be expressed as a function of the three main aims of the process, nevertheless it does not necessarily exist direct quantitative relationship. Pathogens are only indirectly linked with the level of stabilization and their reduction is rather directed at a characterization of biosolids as regards the risks they pose to health. Sewage sludge microbiological populations are extremely sensitive to contamination or contact with microorganisms in the environment which may disturb pathogen monitoring. Although smell is more directly linked with the degree of stabilization reached, its measurement is too subjective for determining the stability of a sludge. The third general aim, the elimination of the potential for putrefaction, is more closely related to the reduction of energy available for microbiological activity which should be the basis to express the stability degree reached [5].

Anyway and despite the widespread use of sludge stabilization processes, no uniform method exists to define or establish the stability level reached. The recommended methods are well known and documented in literature but the lack of standardization plagues many of these recommended tests [6]. There is still no simple, reliable and universally acceptable analytical method for assessing the stability of biosolids, criteria generally varying with the definition of stability in use. One of the approaches with the most scientific basis is that proposed by Hartenstein [7], whereby sludge could be said to be stable when the original solid residues have been converted into non-putrefying odorless humic matter with very slight and very slow degradation. Then, and bearing in mind the basis of stabilization, a parameter for the characterization of stability should be related to the principal process taking place, which is the mineralization of the biodegradable organics of the sludge. From this point of view the widely used loss on the ignition-mineralization index can be justified and experience has ruled it as a parameter for sludge stability [8].

Differential thermal analysis (DTA) and thermogravimetric analysis (TG) are techniques largely untried in sludge characterization. Recently they have been successfully extended to the study of the combustion value of sludges in the same way as for coal or wood [9]. In stabilization process the organic matter in the sludge is transformed to products with a lower total free energy. This fact leads to consider DTA and TG as promising techniques through the control of the energy content of the sludges and the loss of organic matter by oxidation/ignition. These techniques may then be regarded as a sophisticated loss on ignition index related to the fundamental process in the sludge stabilization.

Differential thermogravimetry (DTG) is based on the rate of weight loss and the DTA on the temperature change in a small sample of sludge in comparison with an inert sample while both are submitted to the same temperature program.

DTG profiles make it possible to know for each sample the weight loss which is taking place at each temperature during its combustion. The higher the temperature at which weight loss occur the more resistant and ordered structurally is the organic fraction which is burning. Comparing samples with a different stability degree, temperature changes at which occurs the main weight loss during combustion should be indicative about the organic fraction characteristics. On the other hand, comparing with the starting sample, whenever a weight loss disappears at a temperature at which it was previously observed may indicate that the corresponding organic fraction which was being burnt has been mineralized due to the stabilization process.

The use of DTA enables changes in weight and enthalpy to be measured continuously while the sample is subjected to a programmed heating rate. Any endo- or exothermic reaction undergone by the sample during heating would bring about a difference in temperature between the sample and the reference. Thereby DTA has been shown to make it possible to study the transformation of organic matter in sludge, a transformation characterized by a shift in energy given off to increasingly high temperatures [8].

On the other hand, monitoring the process by TG–DTG would lead one to expect an increase in the combustion residue of the sample, linked with a decrease in the organic fraction of the sludge as the degree of stabilization increases [10].

Hardly any official explicit regulations exist regarding the degree of stabilization acceptable in the treatment of sludge, but those of the US EPA (1993) [11] are probably the most complete as Subpart D of the Part 503 describes certain stabilization requirements for land application of sewage sludge. These requirements make reference to both pathogen and vector attraction reduction (VAR). Assessment of pathogens is an important parameter but only, at best, an indirect indicator of stability so the most closely related requirement in the Part 503 rule to stability, is that of VAR [6]. According to these regulations and from a VAR point of view, for an aerobic biological treatment, at least a 38% reduction in the content of volatile suspended solids (VSS) is required. Nevertheless the concept of VAR equivalency should be considered as a means to promote the development of stability assessment methods for VAR compliance [6].

Even though much of the research effort concerning stability assessment has generated parameters that are useful research tools, rather than practical tools, fundamental research efforts are still needed to enhance our understanding of the decomposition of organic matter and to evaluate practical assessment tools [6].

Having in mind the above said, the main objective of this study was to make an appraisal of the usefulness of DTA and TG–DTG analysis for the characterization of the stability of sewage sludges.

As determination of the stability of biosolids should be based not on one but on several tests contributing complementary information [6] results obtained by TG–DTG analysis have been confirmed by testing total solid and volatile contents and microbiological monitoring throughout the stabilization process carried out on laboratory.

2. Experimental

Solids from different origin were used as starting materials to be stabilized during an aerobic digestion process carried out on laboratory scale. Differences between initial sewage sludge samples should be taken into account as they are going to have an effect on the progress of the stabilization. Sewage sludges employed in this study came from the treatment plants for wastewater from an urban area (AS), from a food industry (KR) and from a milk industry (RO). These three plants are in the province of León (Spain) and biological treatment by means of activated sludge is carried out in them. In all three plants solids produced would usually undergo an aerobic stabilization although those used in this study did not suffered any other treatment than the one in laboratory.

Initially, the organic content of the non-digested sewage sludge samples was 49% for AS, 53% for KR and 92% for RO, expressed in a dry-weight basis.

In each case, laboratory stabilization of the sludges was carried out in 5 l column-shaped aerobic reactors with diffusers for aeration. Air was supplied by means of a compressor and monitored by timers and regulated by rotameters. For each sludge, several reactors were in use simultaneously with different oxygen concentrations, ranging from 1 to 4 mg l^{-1}, this being periodically measured in each one with a WTW-OXI92 oximeter. For the sake of clarity, the data to be compared for the various sludges are for a concentration of 2 mg l^{-1}. Working temperature for all tests was 16–18 °C.

The stabilization process has been monitored for all the experiments by analyzing the total suspended solids (TSS) and the VSS [12] and also by testing Enterobacteriae and coliform bacteria content [13]. Solid matter quantification has been analyzed by sample filtration and by weight loss after drying at 105 °C for TSS and after burning at 550 °C for VSS. Counting of colony-forming units (CFU) have been done after sample dilution and filtration through special filters which later on have been placed and incubated in gel plates with violet red bile glucose (VRBG) for Enterobacters and violet red bile (VRB) for total coliforms.

Thermal analysis was performed with a TA Instruments SDT2960 apparatus registering TG and DTA measurements simultaneously. The gasses outlet of this equipment was connected to a Balzers GSD mass spectrometer, so data concerning gas emission could confirm thermogravimetric results.

To follow-up the stabilization process, apart from the original sample as it was received in the laboratory, a 25 ml homogeneous sample was periodically collected from each reactor. Samples were identified with the corresponding initials followed by the number of days of laboratory treatment; i.e. AS5 denotes the sludge from the urban wastewater treatment plant stabilized during 5 days in laboratory. After each sample was centrifuged at 10,000 rpm for 5 min and the supernatant discarded, the sedimentary fraction was taken for thermogravimetric analysis, which was carried out on dry base to avoid the effect of moisture. The sample (of approximately 30 mg) was subjected to a heating rate of 5 °C min^{-1} to 200 °C, this temperature being kept constant until weight loss ceased, when the dry mass of the sample was recorded. The sample was then heated at 25 °C min^{-1} to 850 °C. In both stages, a flow-rate of 50 $\text{cm}^3\ \text{min}^{-1}$ of synthetic air (composition $21 \pm 1\%$ O_2 and $79 \pm 1\%$ N_2; purity $\geq 99.9994\%$) was maintained, at a manometric pressure of 101 kPa.

3. Results and discussion

Over time, the monitoring of TSS, VSS and microorganisms made it possible to follow the stabilization process. Trends displayed for the three different sewage sludges are shown in Tables 1–3 so it may be observed these parameters decreasing as stabilization advancing.

Taking into account EPA Part 503 regulations [11,14], the required reduction in VSS content of at least 38% has been reached in different times along the stabilization process for the three sewage sludges used in this study (Tables 1–3).

Table 1
Evolution of the stabilization process for the AS sewage sludge

Days of aeration	TSS[a] (mg l^{-1})	VSS[b] (mg l^{-1})	VSS/TSS	VSS reduction (%)	Enterobacters (CFU ml^{-1})	Coliforms (CFU ml^{-1})
0	7000	5600	0.800	0	895E+4	60E+3
2	4800	3500	0.729	38	150E+4	2.5E+3
5	4770	3160	0.662	44	75E+4	<0.1E+3
11	3340	1720	0.515	69	15E+4	<0.1E+3
15	3085	1215	0.394	78	9E+4	<0.1E+3

[a] TSS: total suspended solids.

[b] VSS: volatile suspended solids.

Table 2
Evolution of the stabilization process for the KR sewage sludge

Days of aeration	TSS (mg l^{-1})	VSS (mg l^{-1})	VSS/TSS	VSS reduction (%)	Enterobacters (CFU ml^{-1})	Coliforms (CFU ml^{-1})
0	2610	2400	0.920	0	623E+4	380E+4
2	2360	2050	0.869	15	367E+4	160E+4
5	1630	1390	0.853	42	353E+4	150E+4
11	1180	810	0.686	66	201E+4	120E+4
15	1250	710	0.568	70	154E+4	100E+4

Table 3
Evolution of the stabilization process for the RO sewage sludge

Days of aeration	TSS (mg l^{-1})	VSS (mg l^{-1})	VSS/TSS	VSS reduction (%)	Enterobacters (CFU ml^{-1})	Coliforms (CFU ml^{-1})
0	9000	7700	0.856	0	22E+4	200E+3
2	7200	6000	0.833	22	8E+4	60E+3
7	6670	5500	0.825	29	6E+4	50E+3
11	5600	4600	0.821	40	4E+4	30E+3
16	5500	4420	0.804	43	2E+4	15E+3

After the aerobic digestion carried out in laboratory, sewage sludge organic fraction has been mineralized so final organic content of the samples was 23% for AS, 29% for KR and 75% for RO, expressed in a dry-weight basis.

Although aerobic digestion conditions have been the same, the effect on sewage sludge has been different depending on its own initial characteristics. Taking into account the above parameters, the stabilization along the process has been specially noticeable for AS sewage sludge which may be related to a more easy to digest organic fraction.

3.1. Differential thermogravimetry and differential thermal analysis

The thermo-analytical DTG curves show only the results after 200 °C, thus obviating weight loss associated to moisture loss, which would mask data in the temperature range under study. This does not occur for DTA curves, not taking place an important enthalpy change in this temperature range.

Fig. 1 shows changes undergone by the DTG combustion profiles corresponding to AS along the stabilization process. In the DTG of AS0 it may be observed a shoulder at 275 °C and a first peak at around 320 °C both becoming smaller as the sludges become more stable. On the contrary, around 475 °C, a second peak in combustion rate appears related to AS sludges digestion, this peak increasing in intensity with stabilization (AS5 and AS11) to then decrease and shift to higher temperatures (AS15). Between these two peaks, combustion rate seems to be similar for all samples, no matter their stability degree. Then, two important steps occur during the AS sludges combustion, a marked trend to diminish intensity of the first oxidation weight loss rate peak in favor of the second one in keeping with the stabilization.

From results in Table 1 and specially from VSS reduction it may be observed that the most evident progress towards stabilization for AS takes place after 11 days of aeration. This is in agreement with weight loss results and from DTG profiles it can be noticed that, as stabilization progresses, a change must take place in the structure of the organic matter, causing combustion to be retarded. It has been observed that the more difficult to oxidize is the organic matter the more retarded its combustion, which is in accordance with the peak delay corresponding to more stabilized samples. For AS15 not only a slightly retarded DTG

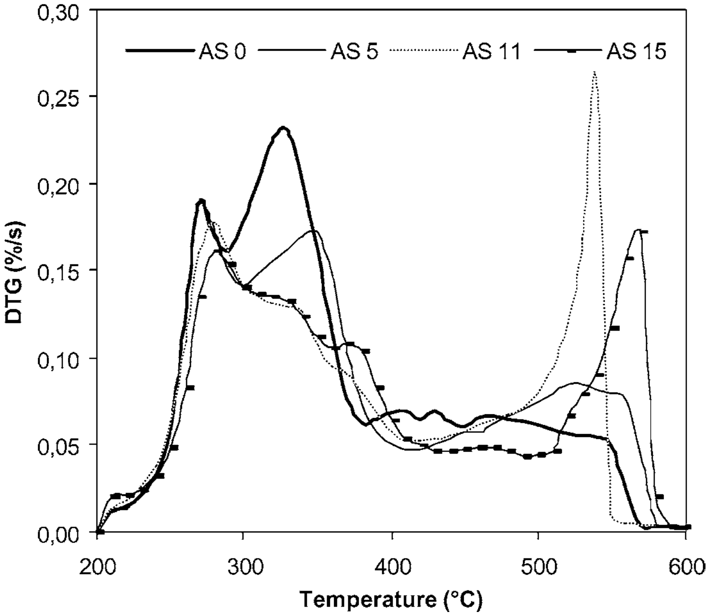

Fig. 1. Evolution of the profile of the weight loss rate in oxidizing atmosphere by days of stabilization treatment for AS sludges (AS0 (━━━); AS5 (—); AS11 (- - -); AS15 (—▪—)).

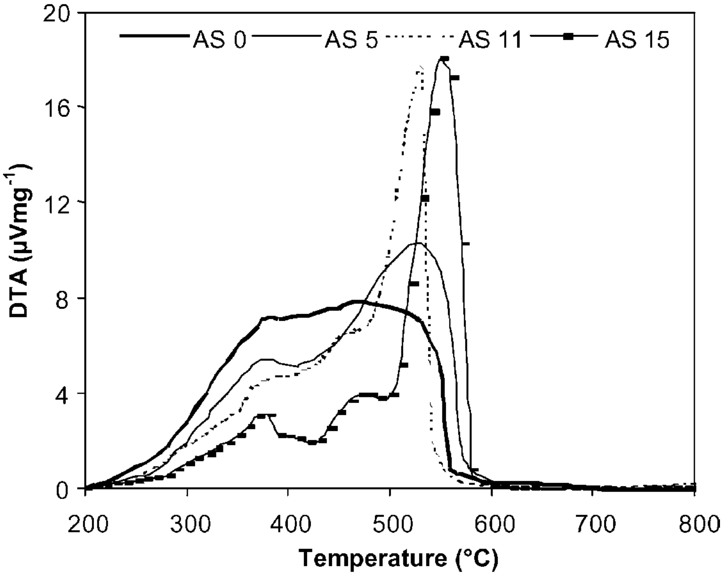

Fig. 2. Evolution of DTA signal in oxidizing atmosphere by stabilization treatment days for AS sludges (AS0 (▬▬); AS5 (—); AS11 (- - -); AS15 (—■—)).

combustion peak but also a lowering has occurred comparing to AS11. This lowering may be related to a weakening in mineralization when this takes place over the most resistant organic fraction from the 11th aeration day to the 15th. Results showed in Table 1 neither show a marked difference between AS11 and AS15.

Regarding changes in enthalpy, DTA corresponding to AS sludges combustions are shown in Fig. 2. Energy release begins, once the moisture has been lost at around 200 °C, the reaction being exothermic to the end at nearly 600 °C. For AS0 sludges, as their organic matter content has not been digested yet, energy release is greater than for the rest of AS samples up to nearly 500 °C. The non-stabilized sludges AS0 may be characterized by its homogeneous combustible content as the highest energy value was reached at around 320 °C to be maintained throughout combustion. At a temperature around 320 °C devolatilization as well as combustion are taking place, which may be seen in the DTG graph (Fig. 1).

As stabilization progresses, samples are not energetically homogeneous any more and AS sludges releases less energy during low-temperature combustion (200–450 °C) than non-stabilized sludges (AS0) because volatile matter is usually the easiest to be digested by micro-organisms and it has been the first to disappear along the process. Together with the AS sludges stabilization progress, the energy profile of combustion becomes more and more exothermic at temperatures above 450 °C, this being related to increasingly complex products of metabolic activity becoming a more important part of the organic content of samples. On the other hand, energy release begins at higher temperatures as the organic matter is being oxidized so, together with the accumulation of complex metabolic products, residual organic matter not yet consumed is structurally more resistant. Fig. 2 shows the same shift in energy release towards higher temperatures as Fig. 1 does in the weight loss rate corresponding to AS sludges combustions, a flattening occurring for AS15 which most exothermic peak along combustion takes place at 570 °C, the same as the corresponding DTG peak. Nevertheless, two steps are not so clearly observed in DTA as in DTG profiles, this indicating not a total energy release parallelism to weight loss during combustion which may have something to do with the organic composition of samples.

Concerning KR sludges from a food industry, two processes may be differentiated during KR samples

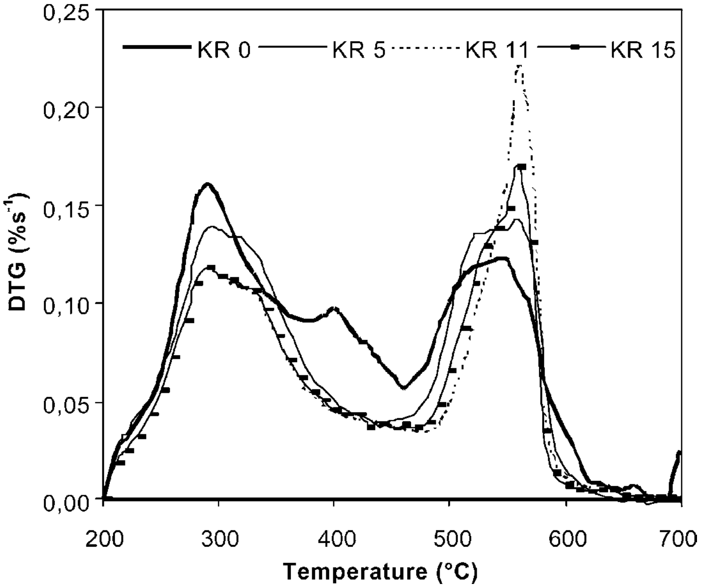

Fig. 3. Evolution of the profile of the weight loss rate in oxidizing atmosphere by days of stabilization treatment for KR sludges (KR0 (———); KR5 (—); KR11 (- - -); KR15 (—■—)).

temperature programmed combustion in their respective DTG curves (Fig. 3). A first peak in weight loss velocity appears at around 280 °C, to diminish in intensity and flatten out along the digestion process, indicating the onset of a more regular rate. Only for KR0 the first weight loss step during combustion appears to be sub-divided into two, the second one corresponding to an organic fraction which, comparing to the whole sample, is rapidly mineralized as it does not appear during KR5 combustion. A second and noticeable weight loss peak occurs between 500 and 600 °C, which in the case of AS sludges did not appear for non-stabilized ones. As stabilization advances, however, behavior at high temperatures is similar to that of AS sludges. Between 390 and 480 °C, the combustion rate for stabilized sludges is low, similar for all cases and lesser than for the initial sludges. Having a different origin and comparing to AS sewage sludge (Table 1), stability reached by KR (Table 2) has been less noticeable. Although initially the starting KR0 sample had a lower solids content than AS0, its VSS/TSS was higher which may have had an effect on the process. On the other hand, DTG combustion profile corresponding to KR0 showed an important weight loss at temperatures over 400 °C which did not occur for AS0. This may correspond to an organic fraction which is not going to be easy to digest and which was already present in the starting sample (KR0).

In Fig. 4, DTA of KR, energy release is greater for non-stabilized sludges (KR0) than for the rest up to 475–500 °C, similar to what happens to AS sludge. Nevertheless, energy release during KR0 combustion does not take place in an only step as for AS0 but a second and more important peak occurs which is consistent with the corresponding DTG combustion profile and with the above-mentioned presence of a quite resistant organic fraction. Energy content in KR0 is associated to the organic fraction which needs a high temperature for undergoing combustion. As stabilization increases, about energy liberation, first, it is concentrated towards higher temperatures, between 500 and 570 °C, where after the intensity of the peak decreases, as may be seen for KR15.

Regarding RO sludge which, as already has been said, had a very high organic content, a DTG peak clearly occurs at 300 °C for RO0 (Fig. 5), decreasing in height as the stabilization process advances. As for the previous sludges, the peak corresponding to the second weight loss step, at a higher temperature, increases in intensity and shifts to higher temperatures as the process continues. Again when a higher stability

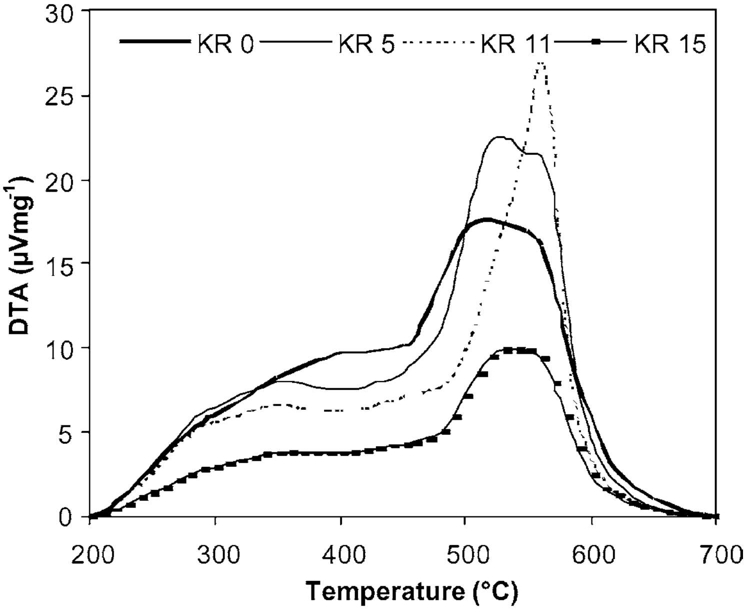

Fig. 4. Evolution of DTA signal in oxidizing atmosphere by stabilization treatment days for KR sludges (KR0 (▬▬); KR5 (—); KR11 (- - -); KR15 (—▪—)).

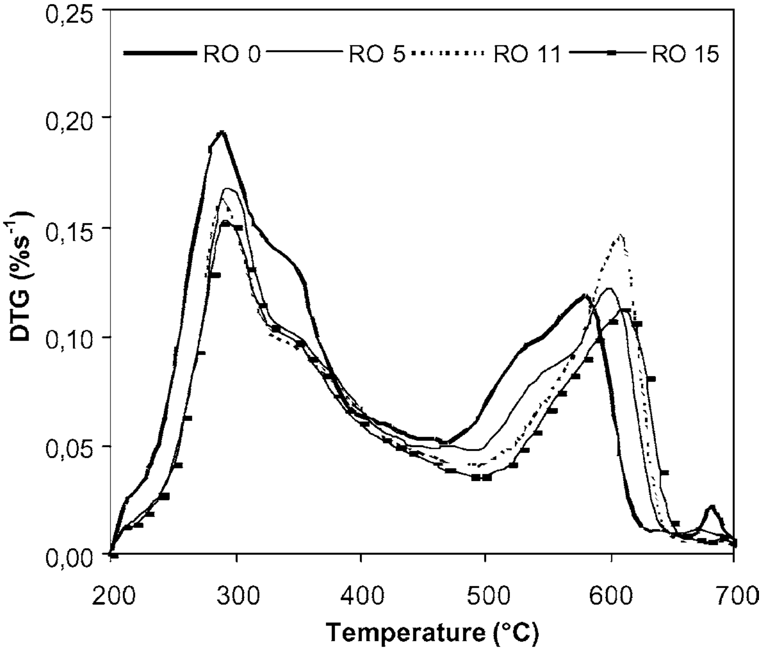

Fig. 5. Evolution of the profile of the weight loss rate in oxidizing atmosphere by days of stabilization treatment for RO sludges (RO0 (▬▬); RO5 (—); RO11 (- - -); RO15 (—▪—)).

is reached, this DTG height peak decreases. Starting RO0 sludge, the same as KR, has also shown two weight loss steps during temperature programmed combustion. Anyway, it should be noticed that the second peak corresponding to RO samples combustion takes place at higher temperature than for AS and/or KR. The weight-loss profile is observed to evolve similarly for RO sludges and the others, although differences should be borne in mind concerning the profiles for the initial samples of each sludge type owing to their different composition. The VSS reduction during RO stabilization (Table 3) has not been so noticeable as for AS and KR. It should be also pointed out that the main VSS reduction took place during the first 7 days of aeration which is consistent with DTG profiles evolution.

In the case of the DTA of the RO sludges (Fig. 6) the initial sample (RO0) as in the cases above, give off more energy than the stabilized samples but, the same as KR0, does not show such an homogeneous combustible content as AS0 does. The stabilization process may have been slightly impeded owing to the starting RO0 characteristics, as there seems to exist an organic fraction which is very difficult to digest under the used experimental conditions so the VSS reduction has been less important than for AS and KR.

As expected DTG and DTA combustion profiles for the samples treated with different concentrations of dissolved oxygen have been similar to those shown in the previously commented figures, the only differences observed being in the length of treatment necessary for obtaining the same results, i.e. greater concentrations of oxygen bring about faster changes.

On the other hand, about TG analyses it should be added that, as it was expected, the weight percentage corresponding to the combustion residue of the three types of sludge studied has showed an increasing trend during the process in keeping with the mineralization which is supposed to be taking place. Also presumably, experiments undertaken with different oxygen concentrations have given off different ash evolution over time, mineralization rate being greater when aeration was more intense. As a matter of an example, evolution of ash content for KR sewage sludge is shown in Table 4.

Mass spectrometry data simultaneous to DTG and DTA curves obtained under oxidizing atmosphere, have been useful to confirm a complete combustion occurring over temperatures around 500 °C. Gases emissions corresponding to the three different sludges employed have followed a similar trend according to their respective DTA and DTG pattern. The profiles of

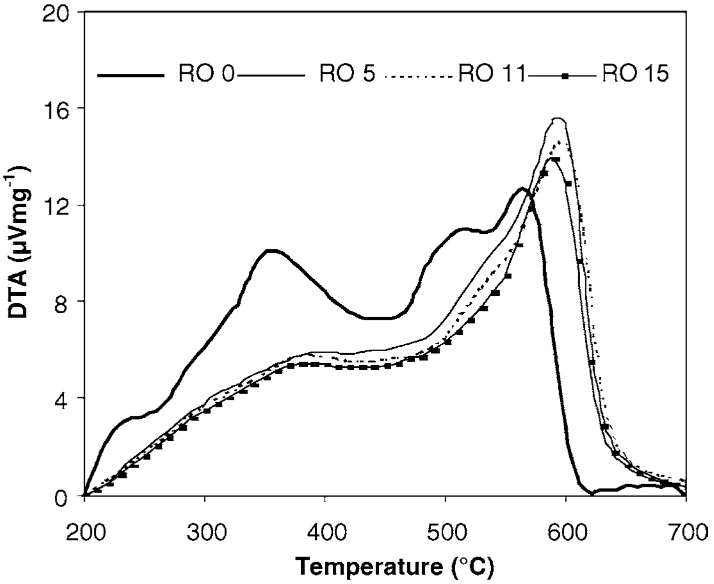

Fig. 6. Evolution of DTA signal in oxidizing atmosphere by stabilization treatment days for RO sludges (RO0 (▬▬); RO5 (—); RO11 (- - -); RO15 (—■—)).

Table 4
Evolution of ash throughout the process of stabilizing KR sludge according to the dissolved oxygen concentration

Days of aeration	1 mg l^{-1} (% ash)	2 mg l^{-1} (% ash)	4 mg l^{-1} (% ash)
0	10.24	10.24	10.24
2	12.80	15.41	16.16
5	15.46	20.36	20.70
11	19.50	26.49	29.14
15	21.49	27.20	30.26

CO_2 emissions during sludges combustion do look nearly exactly like the corresponding DTA curves. Having been observed for all cases that the weight loss shows a slightly different tendency at temperatures bellow 400 °C, it may be said that while a relatively important weight loss is taking place, this is not paralleled by an important energy release neither by a CO_2 emission. This fact proving that at temperatures below 400 °C a non-energetic release of volatile matter takes place.

3.2. Differential thermal analysis: percentage of energy released against temperature

Previous DTA figures have shown a characteristic shift of energy to higher temperatures which could be linked both with digestion of the organic fraction which is most easy to consume by micro-organisms to obtaining energy and also with the metabolic production of increasingly complex humic substances by the micro-organisms which is a typical evolution of the stabilization process [15–19]. Nevertheless, it has been found that integrating DTA curves corresponding to each sample combustion illustrates these results more graphically. The plot of the percentage of area of energy released against temperature may be useful when trying to evaluate stabilization in terms of DTA results so a quantitative value may be established for which the stabilization achieved could be considered suitable. Areas so obtained considering a zero linear baseline are proportional to the energy released, the percentage of the cumulative area having been represented against temperature.

Fig. 7 shows the profiles of the total area percentage at each temperature along the AS sludges temperature programmed combustions. It was observed that for any temperature, the greater the percentage of the area, the lesser the extent of stabilization, so, for example, at 500 °C, it has been released the 70% of all the energy involved in the AS0 combustion process, while for the most stabilized sample (AS15), part of the energy associated to the less resistant organic fraction has disappeared by mineralization so the figure was hardly 40%.

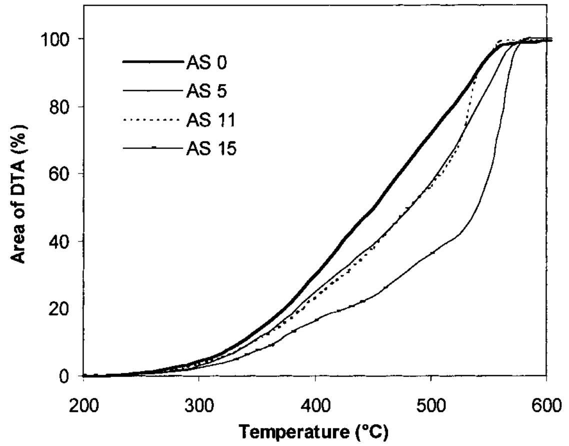

Fig. 7. Evolution of the DTA area percentage against temperature by stabilization treatment days for AS sludges (AS0 (▬▬); AS5 (—); AS11 (- - -); AS15 (—■—)).

In the representation of the percentage of DTA area corresponding to KR (Fig. 8) and RO (Fig. 9) sludges,

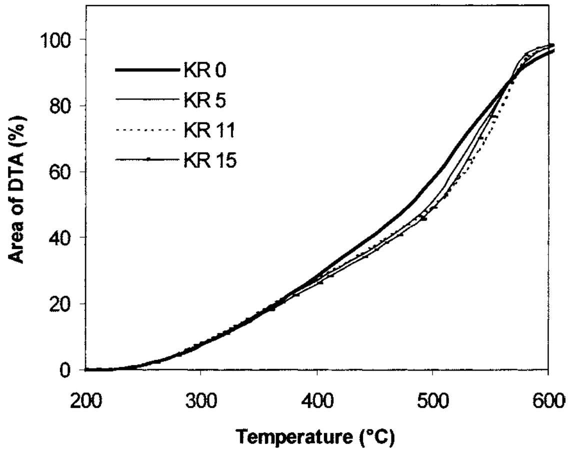

Fig. 8. Evolution of the DTA area percentage against temperature by stabilization treatment days for KR sludges (KR0 (▬▬); KR5 (—); KR11 (- - -); KR15 (—■—)).

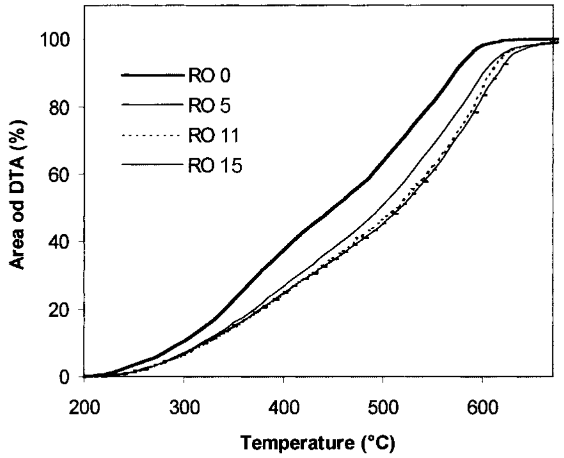

Fig. 9. Evolution of the DTA area percentage against temperature by stabilization treatment days for RO sludges (RO0 (▬▬); RO5 (—); RO11 (- - -); RO15 (—■—)).

the tendency is similar to that of the AS sludges although no so noticeable. This fact must be due to the characteristics of the starting KR0 and RO0 samples which energy content, as it has been already said, seemed to be associated mainly to a quite resistant organic fraction.

On the whole, and taking into account the stability criterion above mentioned (USEPA, 1993) [11,14] it has been found that when the studied sludges have reached a stability acceptable within the scope of USEPA regulations, the DTA area percentage at 500 °C is less than 50%, no matter the sludge neither the dissolved oxygen concentration during its treatment.

4. Conclusions

Stabilization processes are usually said to have followed a normal course by testing a solids content and micro-organisms populations reduction so trends followed by DTG–DTA combustion profiles along stabilization have been checked to be consistent with this reduction. Apart of that, owing to the mineralization process taking place during stabilization treatment, sewage sludge residues from temperature programmed combustion increase as stabilization progresses.

The weight-loss profile produced during combustion in oxidizing atmosphere for non-stabilized sludges may depend on the sample starting characteristics. However, as stabilization advances, no matter the sludge studied, there is a clear tendency to a lower decrease in the rate of weight loss at low temperatures while the rate of weight loss increases with temperature. Finally, when stabilization is quite advanced, the rate of weight loss also start to low at higher temperatures (500–600 °C). This could be related to the organic content digestion by micro-organisms while intense aeration, as for they may feed on the most easy to consume fraction, until shortage force them to consume more resistant organic matter.

As for the DTA, it can also serve as a guide to ascertain the degree of stabilization of biosolids, and it is easy to observe that, in oxidizing atmosphere, for stabilized sludges, the heat released is increasingly less at low temperatures, while above 450 °C they show an increasingly exothermic peak as stability rises. It should be emphasized that no exact parallelism exists between the DTG and DTA profiles as the mass losses occurring between 200 and 400 °C are associated with a low-energy devolatilization, which has been checked by the corresponding mass spectrometry data. It is thus confirmed that as stability increases, less volatile matter is released as this is being consumed by micro-organisms along the digestion process. On the other hand, energy release with advancing stability seems to shift to higher temperatures, in association with the combustion of an organic fraction increasingly difficult to oxidize.

Plotting the percentage of energy released against temperature gives a graph allowing for the analysis of the development of stabilization. From this graph and for each type of sludge, a percentage value may be established for which the stabilization achieved could be considered suitable. It would, however, be necessary to emphasize that the validity of this parameter for each specific sludge demands an unavoidable previous check.

According with the above results, the stability degree reached by sewage sludge during their stabilization treatment may be described in terms of thermogravimetric data, which constitutes not only a very new application of these techniques but also a faster alternative or complementary way for the routine assessment of stabilization. Future research should be made for different kinds of sludges trying to fit data to a kinetic profile. This could allow us to define each sample behavior during its temperature programmed combustion according with the sludge stability degree. It should be tried to correlate this behavior with what is happening to the organic fraction during stabilization, for which masses spectrometry could provide a valuable information.

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