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THE USE OF THERMAL ANALYSIS IN WATER TREATMENT RELATED PROBLEMS

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ABSTRACT

TG, DTG and DSC were used to study and solve water treatment related problems. Wet sludge from a water treatment plant was characterized and drying conditions were determined. A case of possible patent infringement, involving dry chlorine tablets was investigated. Spent activated carbon reactivation difficulties were solved using TG.

INTRODUCTION

Thermal analysis is a grouping of techniques with a wide range of applications. TG has been used to analyse flue-gas scrubber materials (1), to study the reactivation kinetics of activated carbon (2, 3, 4, 5), $CaCO_3$ decomposition (6) and a multitude of other materials. Very often, however, a single technique is not powerful enough to obtain sufficient information on a sample. Therefore, it is necessary to use a combination of different techniques. Paulik et al (6) used a combination of TG, DTG and DTA to analyse clay minerals and $CaCO_3$.

In water treatment, a wide variety of products is encountered and thermal analysis has proven to offer useful techniques to study problems related to water treatment (7). This paper presents the use of TG, DTG and DSC to characterize a lime and clay (suspended solids) containing sludge sample. The same techniques were used to investigate a case of possible patent infringement involving dry chlorine tablets, while TG was used to solve an activated carbon reactivation problems.

EXPERIMENTAL

Sludge, suspended solids and calcium carbonate samples were derived in a dry form (dried at 80°C), from a 300 Ml/d water treatment plant. Prior to analysis these samples were rehydrated to a moisture content of 35%, which corresponds with the moisture content of material at the treatment plant. The sodium silicate sample was received in the unactivated form and activated with diluted hydrochloric acid to pH 9.

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Dry chlorine (HTH) samples (samples A, B and C, where C is the suspected sample), received in a tablet form, were ground to a fine powder (95%<0,045mm) prior to analysis using a ball mill.

Two activated carbon samples from a small water treatment plant (virgin and spent activated carbon) were received in a granular form and part of every sample was ground to a fine powder (grinding to 95%<0,045mm).

All TG work was done on a Perkin-Elmer TGS-2 thermobalance with System 4 controller and Data Station. A Perkin-Elmer DTA 1700 was used for DTA work, while DSC analyses were performed on a Du Pont 9001 thermal analysis system.

RESULTS AND DISCUSSION

Water treatment sludge

The feasibility to fully characterize the water treatment sludge was evaluated using TG. Bedford and Bridle (8) formulated the basic principle for the determination of the powdered activated carbon (PAC) content in a PAC-biosludge mixture using different atmospheres. The feasibility of this principle for the characterisation of a three component water treatment sludge system was evaluated, using nitrogen, carbon dioxide and air as purge gases. DTG maxima were also used so that specific peaks, present in the DTG-curves of both the sludge and one of the single components could be determined. In this way it was possible to identify DTG-peaks which are characteristic for each component. Table 1 presents the different DTG-peak maxima. As the area of a DTG-peak is proportional to the mass loss, this area can in principle be used for the determination of sludge composition. DTA was also evaluated in order to obtain more information on sample composition but was found to be unsatisfactory due to the low sensitivity of the system.

A possible approach to sludge characterisation, using DTG-peaks, is outlined hereafter. In a nitrogen atmosphere a specific peak was found for suspended solids at 280°C although it is uncertain whether this peak is not masking the peak for sodium silicate, which is found at 240°C. A peak which is characteristic for sodium silicate was found between 840 and 850°C using a CO_2 atmosphere while in an air atmosphere a specific peak was found at 780°C for $CaCO_3$. At 295°C, in an air atmosphere, a peak was found for both the suspended solids and the $CaCO_3$. If the peak at 780°C is used to calculate the $CaCO_3$ content then it is possible to determine the suspended solids as a difference between the peaks at 295 and 780°C. In this way it is in principle possible to characterise the water treatment sludge using a set of calibration curves.

Table 1: DTG-peak maxima

Sample	Purge gas	Temperature (°C)			
		T_2	T_2	T_3	T_4
Sludge	N_2	280	470	600	780
	CO_2	300	480	-	840
	Air	295	500	-	780
$CaCO_3$	N_2	-	490	-	800
	CO_2	300	520	675	-
	Air	295	490	-	780
Suspended solids	N_2	280	530	640	-
	CO_2	300	520	675	-
	Air	295	530	650	-
Na_2SiO_3	N_2	240	-	-	850
	CO_2	280	-	690	850
	Air	390	-	-	850

In addition to the characterisation of the sludge, the drying characteristics of the sludge were also required. These characteristics were determined using TG and DSC. From both the TG curve and the DSC plot the maximum drying temperature was determined as 300°C. The energy requirement for drying at this temperature was determined at 85,1 kcal/kg wet (35% moisture) sludge.

Dry chlorine

In this investigation of possible patent infringement the aim was to compare TG and DTG curves (Figure 1 a & b) in order to evaluate the composition of the different samples evaluated. The curves could not be determined on a dry basis because $Ca(OCl)_2$ decomposition occurs at 100°C which is below the drying temperature of 110°C. The TG curves, but more clearly the DTG curves show a characteristic reaction at 430°C for samples A and B which is not found in the curve for sample C. Sample C shows a specific reaction at 140°C which does not occur in samples A and B. These differences between the TG and DTG curves were sufficient to disprove the suspicion of patent infringement.

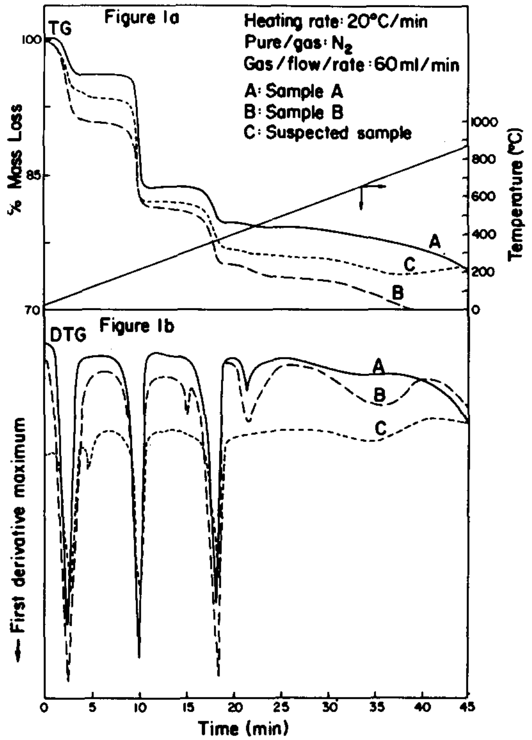

Figure 1: TG and DTG curves of dry chlorine samples

Activated carbon samples

During the actual in-plant thermal reactivation of the spent granular activated carbon only a small fraction of the adsorbate loaded on the activated carbon could be removed. TG analysis of a pulverized sample, however, indicated that a much higher fraction could be removed. Upon inspection, part of the granular activated carbon was found to be covered with a layer of clay like material. This layer was suspected to hinder reactivation by blocking of pore entrances. This blocking prevents the adsorbates to be removed from the pores during reactivation. In order to verify this hypothesis original particulate samples of the virgin, coated and uncoated carbon were analysed (Figure 2, curves A, B and C respectively). The TG curves show that the coated sample has a smaller mass loss than the uncoated sample, indicating that the layer of clay like material hinders removal of adsorbates and thus prevents proper reactivation. These results show that the part of the treatment process preceding the activated carbon adsorption step has to be changed in order to be able to reactivate the activated carbon, i.e. better suspended solids removal is required.

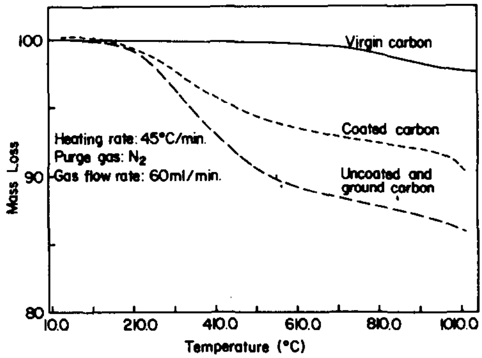

Figure 2: TG curves of activated carbons

CONCLUSIONS

TG, DTG and DSC are very useful tools to investigate such water treatment related problems as: sludge characterisation and drying, product evaluation and evaluation of spent activated carbon regeneration.

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