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INVESTIGATION OF CONCRETE ADDITIVES BY EVOLVED GAS ANALYSIS

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An evolved gas analysis method measuring the organic gases and vapours was applied in the investigation of concrete additives. Several commercial superplasticizers and solid cement mortars containing one of them in different concentrations were measured. EGA curves offer some information for the identification of additives, however, the thermal behaviour of the additives in the substantial form and in the mortar is different. The area under the EGA curve and characteristic intensities of the signal may be applied in the quantitative determination of the additives in cement mortars.

Concrete additives are frequently used to change the viscosity of the cement slurry, to accelerate bonding, to form gas bubbles in the concrete, etc. The additives are usually organic compounds or mixtures prepared from by-products of different industries (e.g., paper making). They are applied is small concentrations, usually below 1% of the cement.

Several methods have been applied to detect and measure the additives in cement mixtures, mortars and concretes: physical tests, titrimetry, chromatography, IR spectroscopy, mass spectrometry, etc. These are listed in detail in a previous paper [1]. However, in the majority of the suggested methods, the instrumental measurement is preceded by some sort of extraction or separation on a column, rendering these procedures rather long.

Among the thermoanalytical methods, which do not necessitate separation steps prior to the analysis of concrete additives, applications of DTA [2–6], DSC [7], and some combined techniques [8–11] have been reported.

The present work deals with the possibility to use evolved gas analysis to identify and measure concrete additives. For our earlier results see ref. [1].

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Experimental

Several commercial superplasticizers (Table 1) and cement mortars containing one of them in different concentrations were measured by means of DuPont 916 Thermal Evolution Analyzer. In this apparatus, the sample is situated in a temperature programmed furnace, and the volatile products are swept into a hydrogen flame ionization detector. This technique is suitable to investigate the evaporation and decomposition of organic matter in the presence of an inorganic matrix.

In the EGA measurements, the samples were heated with 16 deg min^{-1} in flowing nitrogen. The evaluation of the curves was based on the characteristic temperatures and signal intensity.

Table 1 The investigated superplasticizers (MFS: melamine-formaldehydesulfonic acid; NFS. naphtalenesulfonic acid; LS: Ligninsulfonic acid)

	Commercial name	Main components
a	Melment L 10	MFS-NA
b	Viskoment V	MFS-NA + LS-Na
c	Fliessmittel 78	MFS-NA + LS-Na
d	Sikament	NFS-Na
e	Daracem 190 HC	NFS-Na + LS-Na
f	Melment F 103	Na stearate + Na carbonate

Results and discussion

The EGA curves of the superplasticizers are shown in Fig. 1. Visibly, these curves provide information for the identification of the additives in the substantial form. However, it should be noted that the composition of the products is not exactly defined, and the thermal behaviour of different batches of the same quality may be somewhat different.

EGA curves of the basic mixture (cement mortar without additive) and three samples with different amounts (0.038–0.151%) of Fliessmittel 78 are presented in Fig. 2. The technological range is covered by the concentrations of these samples. Note that both sample mass and sensitivity had to be drastically increased, as compared to the runs on additives in the substantial form.

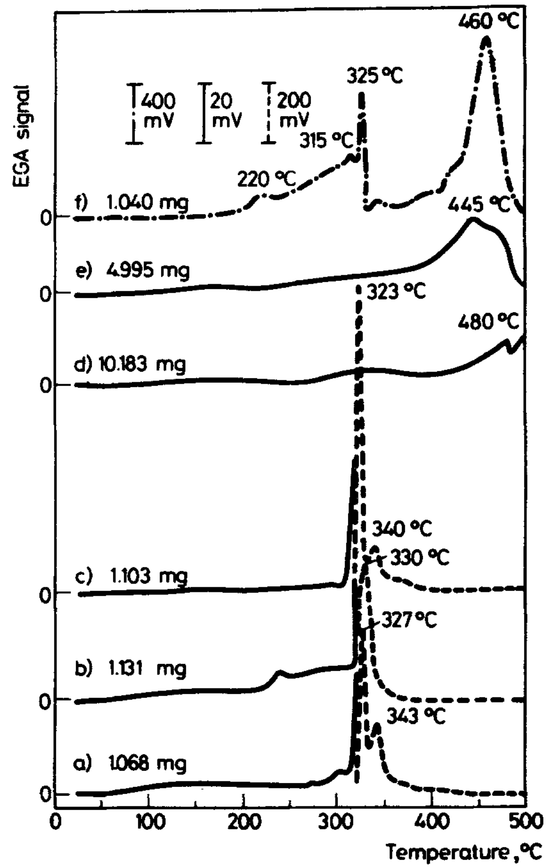

Fig. 1 EGA curves of superplasticizers. Curves a–f correspond to the materials listed in Table 1.

Table 2 Some characteristics of the EGA curves of cement mortars containing Fliessmittel 78. The results of five parallel runs (A: total area under the curve; m: sample mass; S_{II} and S_{III}: EGA signal at peak maxima II and III, respectively)

Sample	Additive content, %	A/m, cm/mg	S_{II}/m, mV/mg	S_{III}/m, mV/mg
a	–	1.21	0.039	0.053
b	0.038	1.46	0.073	0.051
c	0.076	1.59	0.077	0.060
d	0.151	2.18	0.132	0.068

The basic mixture was not completely free of organic material either (see curves of Fig. 2), so the mortars show the effect of the basic mixture and the additive together. Moreover, comparing the curves of Fig. 2 to curve c of Fig. 1 points to the fact that the decomposition of the additive in the substantial form and in the mortar is different.

For quantitative purposes, the total area under the EGA curve and the intensity of peaks II and III may be suitable, as peaks I and IV are firstly related to the basic mixture (see Fig. 2). The results of parallel runs are summarized in Table 2; all the three demonstrated parameters show an increasing trend as a function of the relative amount of the additive.

Thus, the method is expected to be applicable for the determination of organic additives in cement mortars and concrete. The statistical evaluation of the results and the behaviour of mortars containing other additives will be reported in a subsequent paper.

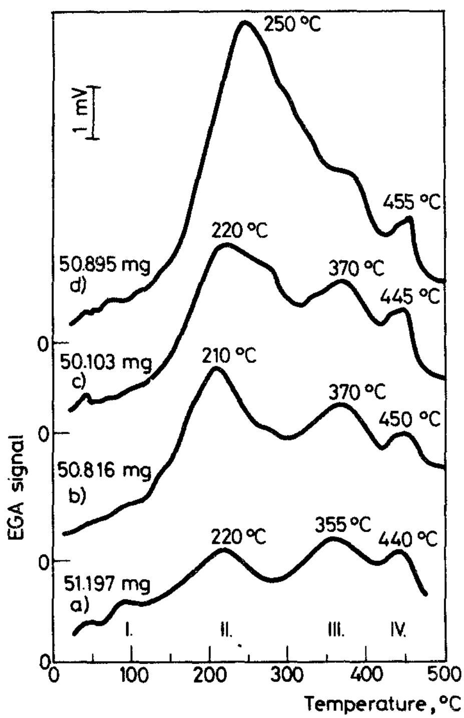

Fig. 2 EGA curves of cement mortars containing Fliessmittel 78. The relative amounts of the additive and some characteristics of the curves are listed in Table 2.

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Zusemmenfassung – Eine thermo-gasanalytische (EGA-) Methode zur Messung organischer Gase und Dämpfe wurde zur Untersuchung von Betonzusätzen verwendet. Einige kommerzielle Superverflüssiger sowie erhärtete Zementmörtel, die einen derselben in unterschiedlichen Konzentrationen enthalten, wurden untersucht. Die EGA-Kurven bieten einige Informationen zum Nachweis der Zusätze, deren Verhalten in substanzieller Form und im Mörtel jedoch differiert. Die Fläche unter dem EGA-Effekt und charakteristische Intensitäten des EGA-Signals können zur quantitativen Bestimmung der Zusätze im erhärteten Zement genutzt werden.

РЕЗЮМЕ — Для исследования различных добавок-присадок был использован метод анализа выделяющегося газа (АВГ). Были измерены несколько продажных пластификаторов и твердых цементных растворов, содержащих в различной концентрации один из пластификаторов. Кривые АВГ дают некоторую информацию относительно идентификации добавок. Однако, термическое поведение добавок в их исходной форме и в строительных растворах совершенно различное. Площадь кривой АВГ и характерные интенсивности сигнала могут быть использованы для количественного определения добавок в цементных растворах.