*Thermogravimetric Determination of Water in Household Liquid Detergents

Dhoaib Al-Sammerrai and **Suham Al-Madfai**
Petroleum Research Centre, PO Box 10039, Jadiriyah, Baghdad, Iraq

Thermogravimetric analysis has been applied successfully in determining the amount of water constituent in household liquid detergents. Results were comparable to those obtained from the standard methods of testing.

The simple, rapid technique is potentially useful and can be considered as a supplementary tool to the conventional methods of analysis.

Household liquid detergents are products obtained by dissolving surface active agents, property improvers and additives in water, which forms the bulk of the liquid phase (1). The surface active agents are compounds composed mainly of the sodium salts of alkyl benzene sulfonates and alklyl sulfates (2) and usually constitute about 20 to 25% by mass with additives and water making up the rest of the composition.

Analysis (3) and determination of water or surface active ingredients in liquid detergents is of importance in knowing the composition of competitive products, for referring purposes and quality control.

The methods most commonly used to determine the water content of liquid detergents are ASTM method D 460, Sampling and Chemical Analysis of Soaps and Soap Products (sections 10-15) (4), which describes an oven and distillation method for water determination, and determination of water by the Karl Fischer technique (5). These methods have their own equipment, sample size and other requirements.

Recently, thermoanalytical methods such as differential thermal analysis (DTA), differential scanning calorimetry (DSC) and thermogravimetry (TG) have been used extensively (6-8) for rapid and accurate analysis of different types of organic and inorganic compositions.

In this paper, a thermogravimetric technique is described for the quantitative determination of water in household liquid detergents containing only anionic type surfactants. Results were compared with those obtained from the standard methods of testing.

The simple procedure used can be considered as a supplementary tool to the conventional methods of analysis.

EXPERIMENTAL

Five commercial household liquid detergents were obtained from different sources; some of their properties are shown in Table 1.

TABLE 1

Some Properties of the Household Liquid Detergents Studied

Sample	Viscosity, centipoise at 30 C	Bulk density	pH
A	287	1.02	6.9
B	295	1.02	7.0
C	310	1.03	7.1
D	295	1.02	7.0
E	300	1.03	6.9

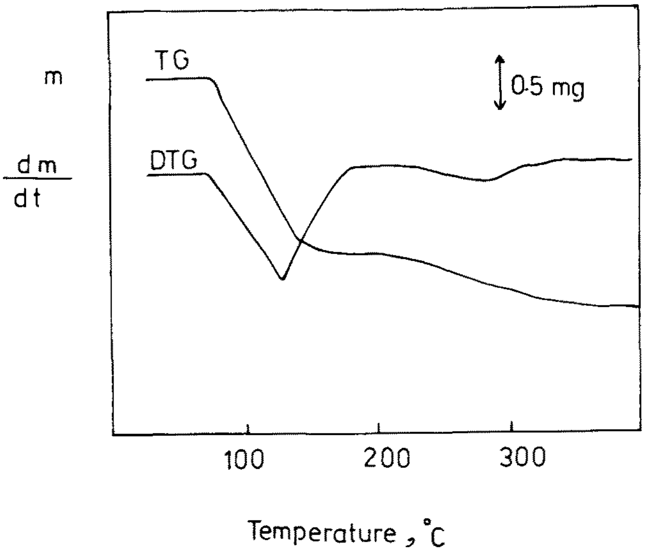

FIG. 1. Representative TG and DTG thermograms for a household liquid detergent.

Thermogravimetric (TG) and differential thermogravimetric (DTG) measurements were carried out on a Heraeus TA 500 thermal analyzer. TG and DTG curves were recorded simultaneously by placing a sample weighing 10-15 mg in a platinum crucible and heating at a rate of 20 or 10 C/min in an atmosphere of nitrogen gas flowing at 30 cm^3/min. The homogeneity of the samples taken was checked from the reproducibility of the thermograms.

Determination of the total water content present in the detergents was performed according to ASTM method D 460 and by the Karl Fischer technique. All measurements were performed in triplicate.

RESULTS AND DISCUSSION

Figure 1 shows representative TG and DTG traces obtained between room temperature and 400 C for the liquid detergents studied. The mass losses recorded in the inert atmosphere of nitrogen gas correspond mainly to the volatilization of the detergent composite.

At a heating rate of 20 C/min, two major mass loss steps were identified. The first, with an initial,

TABLE 2

The Water Content (wt %) of the Liquid Detergents As Determined by Different Methods[a]

Sample No.	Thermogravimetry	Oven Method ASTM D 460	Distillation ASTM D460	Karl Fischer Technique
A	77.0	75.5	76.5	77.0
B	77.5	76.0	76.5	77.0
C	76.0	75.0	76.0	76.5
D	77.0	76.0	76.5	76.5
E	76.5	75.0	76.0	76.5

[a] Average of three determinations.

maximum and final temperature of transition (from DTG trace) of 75, 130 and 175 C, respectively, corresponds to the volatilization of the water constituent and the ethyl alcohol incorporated in the composition in concentrations ranging between 1 and 2% depending on whether the batch is manufactured in summer or winter. The remaining step, which proceeded at > 50 C higher, is related to partial degradative volatilization of the surface active and other ingredients with initial and final transition temperatures beginning and ending at about 240 and 350 C, respectively. It is well known that alkali metal salts of alkyl sulfonate and sulfate derivatives begin decomposing at ~250 C (9).

The mass losses and transition temperatures obtained on evaluation of the thermograms when the determinations were performed at a slower heating rate of 10 C/min were similar to those of the faster heating rate (20 C/min).

Results of the triplicate measurements for the samples studied at the two different heating rates showed an estimated repeatability limit of within ± 1%.

Table 2 shows the amount of water in weight % present in the studied liquid detergents which were estimated from the TG traces of the first mass loss step. Also in the table are the values of the water content of the same detergents, which are obtained according to ASTM method D 460 (oven and distillation procedures) and by the Karl Fischer technique.

From the results obtained, it appears that the performance of the methods mentioned for the determination of water content in liquid detergents will yield results of comparable accuracy after compensating for the ethyl alcohol present, which will volatilize with the water depending on the testing procedure used.

It should be pointed out that the difference of > 50 C between the end of volatilization of water and the start of the degradative volatilization of the surface active constituents of the liquid detergents is adequate for rendering the present technique (TG) applicable, as this will ensure avoiding interferences from effects such as thermal stability, molecular association and other possible interactions.

By using this technique a sample can be assayed in about 25 min when choosing the faster heating rate; furthermore, on neglecting the transition following the water volatilization, the experimental running time could be reduced by about one-half.

Factors weighing heavily in favor of using thermogravimetry are shorter time, smaller sample size and simplicity of the technique. Also, upon expanding the temperature program, the thermograms obtained can be used to study other properties of liquid detergent compositions, e.g. thermal stability.

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