Determination of Water Content of Granulated Detergents

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ABSTRACT AND SUMMARY

A quick analytical procedure was described for the determination of water content of granulated detergents. A constant weight loss of sample was obtained within 5 min by microwave drying. The weight loss agrees with the water content by the modified distillation method. The coefficient of variation was 0.7%.

INTRODUCTION

Water content of granulated detergent can be measured by different common methods such as the oven drying method (1), the distillation method (2,3), and the Karl Fischer method (4). However, the accurate determination of water content of a granulated detergent has been the subject of several analytical investigations (5), and is a continuing problem.

The oven drying method is one of the most widely used techniques because of its simplicity. The weighed sample is heated in an oven at atmospheric pressure, and the total weight loss is taken as a measure of the water in the sample. The method does not differentiate between water and other volatile materials. Besides, some hydrated inorganic salts are quite stable even at high temperatures and do not lose all the water of hydration upon heating. Thus, the direct determination of water in granulated detergent is impeded. The distillation method is based upon a heterogeneous azeotropic distillation with xylene or other solvents immiscible with water. A sample is refluxed in the solvent, and the aqueous phase is measured volumetrically. The distillation method gives water contents which are closer to the actual water content than can be obtained by the oven drying method. However, the reproducibility of the results by the distillation method is rather poor because of the following reasons. The calculation of water content is based upon inaccurate visual reading of a small volume (usually a few milliliters) of water in a graduated collecting tube. Errors are often caused by the adherence of water droplets to the side of the condenser and receiver, unless the equipment is cleaned very carefully in each determination. Karl Fischer titration, in general, is the most widely applicable method for accurately determining the water contents of various samples. This method, however, cannot be applied directly to the determination of water in granulated detergents since the latter frequently contain sodium carbonate which reacts with the Karl Fischer reagent. Water of hydration of some salts in detergents dissolves very slowly in the solvent and makes the Karl Fischer titration difficult. As a quick method for determining the water content of granulated detergent, infrared lamps have been used as heat sources for the rapid drying of samples, and some moisture testers (6) of this type are commercially available. Recently, microwave radiation has been widely used for drying materials such as wood, rubber, textiles, and polymers for industrial purposes. Microwave ovens are now common apparatus for general heating and drying operations in the laboratory (7). This paper describes a quick method for determining water content of granulated detergent by microwave drying. The advantages of microwave drying are: (a) heat is generated throughout the sample, and drying takes place internally and homogeneously; (b) the drying rate is very fast and evaporation of water occurs quickly. A commercially available apparatus, a microwave oven combined with a built-in electronic balance, was used. By drying a detergent sample under defined conditions, weight loss corresponding to the water content by a modified distillation method (D-KF method) was obtained within 5 min.

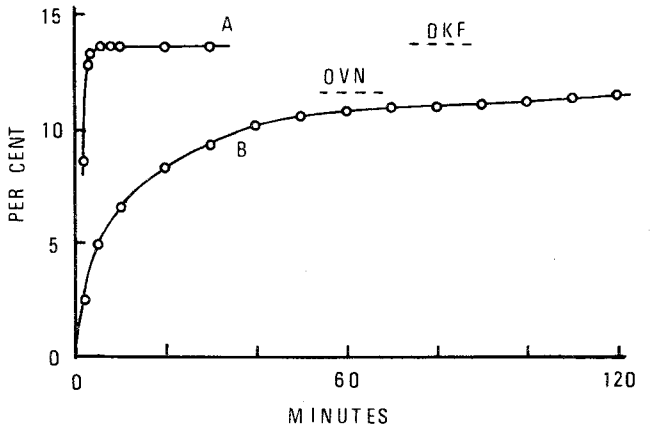

FIG. 1. Weight loss of granulated detergent by microwave drying. Microwave heating: continuous–initial 3 min, intermittent–20 sec on and 10 sec off, sample: 5 g; curve A: 20 ml of water added; curve B: no water added. DKF, OVN: water content by D-KF and oven drying methods.

EXPERIMENTAL PROCEDURES

Reagents

Reagent grade or primary standard grade chemicals were used unless otherwise stated. Karl Fischer reagent was purchased from Mitsubishi Chemical Industries Ltd. (Tokyo, Japan).

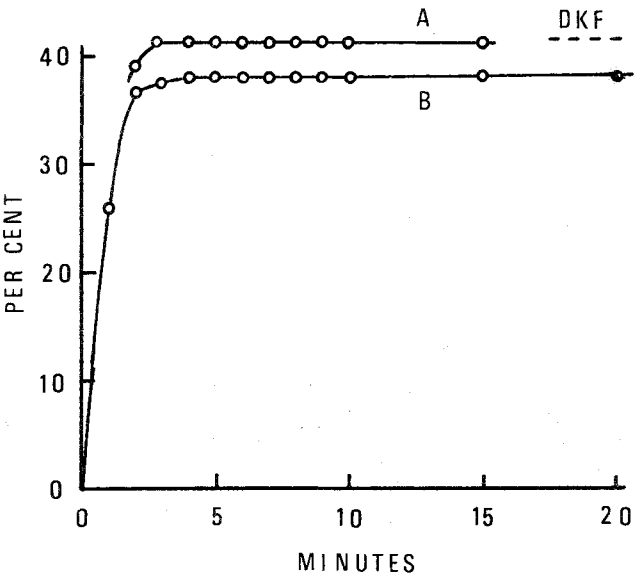

FIG. 2. Weight loss of detergent slurry by microwave heating. Microwave heating: continuous–initial 3 min, intermittent–20 sec on and 10 sec off, sample 5 g; curve A: 20 ml of water added; curve B: no water added, DKF: water content by D-KF method.

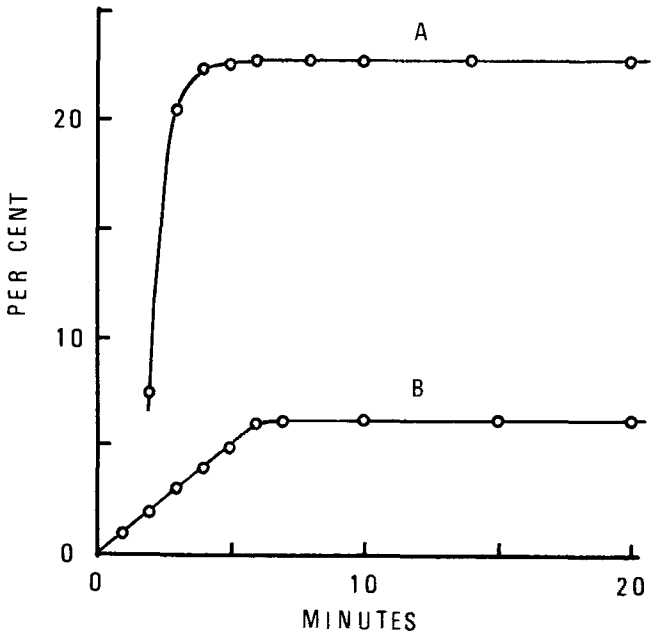

FIG. 3. Weight loss of $Na_5P_3O_{10}\cdot 6H_2O$ by microwave drying. Microwave heating: continuous–initial 3 min, intermittent–20 sec on and 10 sec off; curve A: 20 ml of water added; curve B: no water added.

TABLE I

Determination of Water Content

Sample	Microwave drying	D-KF	Oven drying
Granulated detergent	%	%	%
A	13.6	13.7	11.6
B	12.0	12.1	10.7
C	13.2	12.9	11.0
Detergent slurry			
A	41.3	41.6	39.1
B	45.2	45.6	42.7
C	43.2	42.7	39.8
D	38.8	38.1	36.9

Apparatus

Microwave Moisture Tester (Model TMM-5501, Toshiba Electrics, Tokyo, Japan) was used. The instrument consists of two major parts, a domestic microwave oven and an electric balance with a digital readout. The microwave oven has a frequency range of 2450 ± 50 MHz and output of 400 watts at 105 volts. The standard distilling apparatus described in ASTM D1568-68 was used with the following slight modification of the graduated receiver. A two-way stopcock was connected at the bottom of the 10 ml graduated receiver to drain the condensate conveniently. The apparatus should be cleaned and dried before use.

Microwave Drying Method

Place about 5 g of detergent sample on a ceramic glass weighing pan and record the sample weight. The sample of detergent is slurried by adding 20 ml of deionized water. Start microwave heating. After an initial 3 min heating period turn the unit on for 20 sec and off for 10 sec periodically. All the procedures above can be performed automatically by the automatic timer of the instrument. Constant weight is obtained by heating the sample in the manner for 5 min. Calculate the weight loss of sample based on the difference of weight before and after drying.

D-KF Method

In the D-KF method, distillation was used strictly as a separation step. Karl Fischer titration was employed for the determination of water in the condensate.

A quantity of sample containing about 1 g of water was weighed accurately into a 1000 ml distilling flask. The sample was suspended in 200 ml xylene with a few boiling chips. The flask and receiver were connected to the condenser. About 30 ml of xylene was poured down the condenser tube to cause a slight overflow through the side tube. Heat was applied directly to the flask with an electric heater. The temperature was raised to boiling, and the condensate was collected in the receiver. The heat was adjusted to maintain an appropriate reflux. After most of the water was distilled over (usually 1 hr), the distillation rate was increased. Distillation was continued until the water level in the receiver remained constant for more than 30 min. When the distillation was completed (about 2 hr), the receiver was detached. The two-phase condensate in the receiver was drained through the two-way stopcock into a 100 ml volumetric flask. Three portions of 10 ml methanol were used to wash down any droplets of water adhering inside the condenser and receiver. They were combined with the condensate in the 100 ml volumetric flask. Additional methanol was used to dilute to the mark. The ternary mixture of xylene-methanol-water in the flask was homogeneous as it contained a large volume of methanol. Two milliliters of the ternary mixture were pipetted into the titration vessel of Karl Fischer titration for the determination of water. Blank determinations were made in the same manner.

RESULTS AND DISCUSSION

Control of Microwave Radiation

In the determination of the water content of detergents by microwave heating, moderate heating by an on-off cycle was important for complete and successful drying. Continuous heating under microwave radiation caused charring of detergent sample. The detergent sample began to burn, depending upon the thickness of sample layer on a weighing pan, sometimes between 5 and 20 min after starting the microwave radiation. This happened because organic materials in the sample also absorbed microwave radiation, especially at high temperatures. Once a little localized temperature increase occurred, the density of the microwave radiation increased at that spot causing a rapid temperature rise, and the temperature rise accelerated until the sample charred. To overcome the difficulty, moderate heating by intermittent microwave radiation was employed.

The effect of added water to the sample is shown in Figure 1. A sample of granulated detergent exposed to intermittent microwave radiation without addition of water lost weight fairly rapidly for the initial 30 min and then kept on losing slowly (curve B). After 2 hr of intermittent microwave radiation, the weight loss reached the level attained by a conventional oven drying method. When the granulated detergent sample was slurried by adding 20 ml of deionized water, the slurry lost weight rapidly and attained constant weight in less than 5 min. This was probably due to the fact that hydrated salts in the sample were dissolved in the added deionized water, and the water of hydration was converted to free water which was evaporated quickly upon microwave heating. The constant weight agreed very well with the water content determined by D-KF method as illustrated in Figure 1. When the more than 20 ml of water was added, a few more minutes were required to reach the same constant weight (not shown in Fig. 1). A similar example is shown in Figure 2 where a detergent slurry sampled during the manufacturing process was analyzed by microwave drying. The sample originally contained about 40% water. The addition of water was also

necessary for the slurry sample in order to obtain a weight loss which agreed with the water content by the D-KF method. The behavior of water of hydration was demonstrated clearly in Figure 3 where the weight loss of tripolyphosphate hexahydrate was plotted against the period of microwave heating. Curve A representing percent weight loss for the slurried sample shows constant weight after 5 min, while curve B representing no addition of water indicates incomplete drying. The values for constant weight loss of curves A and B correspond to a hydration of 6 H_2O and 1.7 H_2O, respectively. Thus, the addition of water was very effective in reducing the time required for complete microwave drying of samples containing hydrated salts.

Precision

The water content of a granulated detergent was determined repeatedly by microwave drying and D-KF methods. The mean values were 13.6 and 13.7% for microwave drying and D-KF methods, respectively. The coefficient of variation in both methods was 0.7%, which is obviously smaller than the typical value of 2-3% in a conventional distillation method. Several plant samples were analyzed by microwave drying, D-KF, and oven drying (105 C, 2 hr) methods. The results are compared in Table I. For all the samples, water contents by the D-KF method and microwave drying method agree well with each other, but the oven drying method yields lower results by roughly 2%. The difference may be ascribed to incomplete removal of water of hydration by the oven drying method.

Interferences

Thermally unstable compounds in the sample will decompose during the determination. For example, sodium percarbonate yields water upon heating.

$$2Na_2CO_3 \cdot 3H_2O_2 \rightarrow 2Na_2CO_3 + 3H_2O + 1.5O_2$$

This thermal decomposition occurs in the oven drying method as well as in microwave drying and D-KF methods. Sodium bicarbonate will likewise undergo thermal decomposition.

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