
EXPERIMENT 3 DETERMINATION OF ASH AND ITS CHARACTERISTICS

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3.1 INTRODUCTION

Ash content of foodstuffs represents inorganic residue remaining after destruction of organic matter. Acid-insoluble ash is a measure of sand and other silicious matter present. High ash content and/or low alkalinity of the ash in some cases could be due to the presence of adulterants.

Objectives

After studying and performing the experiments, you should be able to:

- determine the total ash content in
 - water-soluble ash;
 - acid-insoluble ash; and
 - the alkalinity of the ash in food products.

3.2 EXPERIMENT 3a: DETERMINATION OF TOTAL ASH

3.2.1 Principle

When a sample of a food material is ashed around 525°C, the organic matter present is decomposed and expelled. The residue remaining is the mineral matter. The ash content is expressed as per cent weight by weight.

3.2.2 Requirements

Equipment and Apparatus

Silica dish (100ml cap.)
Chemical balance, 1mg sensitivity
Hot plate or burner
Muffle furnace
Desiccator (with an active desiccant)

Precautions

Do not transfer the hot silica dish directly from the muffle furnace to the desiccator. Cool to 100°C and transfer to desiccator.

3.2.3 Procedure

Note the tare weight of three silica dishes. Accurately weigh 5 g of sample into each. Char the material carefully on a burner or hot plate and transfer the dishes to a muffle furnace and ash at a temperature of around 525°C until a white ash is obtained. Moisten the ash in dishes with water. Dry on steam bath and on hot plate and re-ash at 525°C. Cool in a desiccator and weigh. Reserve the ash in one dish for determination of water-soluble and water-insoluble ash, in second dish for acid-insoluble ash, and the ash in the third dish for determining alkalinity of ash.

3.2.4 Observations

Weight of silica dish	= W ₁	= ----- g
Weight of the silica dish with sample	= W ₂	= --- --- g
Weight of the silica dish with ash	= W ₃	=----- g

Calculation

$$\text{Total ash (\% in the sample)} = \frac{\text{Weight of ash}}{\text{Weight of sample}} \times 100 = \frac{(W_3 - W_1)}{(W_2 - W_1)} \times 100$$

If the ash content has to be calculated on dry weight basis:

$$\text{Total ash content (\% on dry weight basis)} = \frac{(W_3 - W_1) \times 100}{(W_2 - W_1) \times (100 - M)}$$

Where, M is the moisture content (%) of the sample in percent by weight (determined as in experiment 2)

Expression of Results

Ash Content = % by weight or % by dry weight

3.3 EXPERIMENT 3b: DETERMINATION OF WATER-SOLUBLE AND WATER-INSOLUBLE ASH

3.3.1 Principle

When the total ash obtained from the above experiment is boiled in water, part of the ash dissolves (water-soluble ash) and part remains as insoluble ash. The insoluble ash can be estimated gravimetrically. The difference between total ash and water-insoluble ash gives water-soluble ash.

3.3.2 Requirements

Same as used for total ash

Reagents/chemicals

Whatman No. 41 filter circles
Red litmus paper

3.3.3 Procedure

Transfer the ash from one of the three silica dishes, with the aid of about 20 ml distilled water, into a beaker. Cover with a watch glass and boil for 5 min. Filter through an ashless filter paper (Whatman No. 41). Wash the entire residue with hot water until the filtrate no longer turns red litmus blue. (Reserve the entire filtrate for the determination of alkalinity). Dry the ashless paper with residue (water insolubles) in the same silica dish and transfer to muffle furnace and ignite at 525°C for 2 hours. Cool in a desiccator and weigh.

3.3.4 Observations

Weight of silica dish No.1 = W_1 --- g

Weight of the silica dish with sample = W_2 --- g

Weight of the silica dish with total ash = W_3 --- g

Weight of the silica dish with water-insoluble ash = W_4 --- g

3.3.5 Calculations

Water-soluble ash (%) = $\frac{\text{Total ash} - \text{water-insoluble ash}}{\text{Weight of sample}} \times 100$

$$= \frac{(W_3 - W_1) - (W_4 - W_1)}{(W_2 - W_1)} \times 100 = \frac{(W_3 - W_4)}{(W_2 - W_1)} \times 100$$

$$\text{Water-soluble ash (\% on dry wt.)} = \frac{(W_3 - W_4) \times 100 \times 100}{(W_2 - W_1) \times (100 - M)}$$

Where, M = Moisture % of sample.

3.3.6 Results

Water-soluble ash = % by weight on dry basis.

3.4 EXPERIMENT 3c: DETERMINATION OF ACID-INSOLUBLE ASH

3.4.1 Principle

Acid insoluble ash refers to the portion of ash, which does not dissolve in 1:2.5 HCl under the experimental conditions. This can be estimated either from the total ash (as obtained in Experiment 3a) or water-insoluble ash (as obtained in Experiment 3b).

Preparation of Sample

Use the ash of second disk obtained from the Experiment 3a.

3.4.2 Requirements

Apparatus

Same as used for total ash

Chemicals/ reagents

Hydrochloric acid - dilute with distilled water (1: 2.5)

Whatman No. 41 filter circles

Blue litmus paper

Precautions

Do not inhale the vapours of Conc. Hydrochloric acid.

3.4.3 Procedure

To the ash of the second silica dish, add 25ml of hydrochloric acid, cover with a watch glass and boil gently for 5 min. Filter through ashless filter paper. Wash the entire residue with hot water (> 85°C) until the filtrate no longer turns blue litmus paper red. Dry the ashless paper with the residue in the same dish and transfer to Muffle furnace and ignite at 525°C for 2 hrs. Cool in a desiccator and weigh.

Alternatively, water-insoluble ash as obtained in Experiment 3b can also be used.

3.4.4 Observations

Weight of silica dish No. 2 = W_1 = --- g

Weight of silica dish with sample = W_2 = --- g

Weight of the silica dish with ash insoluble in acid = W_3 = --- g

3.4.5 Calculations

$$\begin{aligned}\text{Ash insoluble in acid (\%)} &= \frac{\text{Acid-insoluble ash}}{\text{Weight of sample}} \times 100 \\ &= \frac{(W_3 - W_1)}{(W_2 - W_1)} \times 100\end{aligned}$$

$$\text{Ash insoluble in dilute HCl (\%)} \text{ on dry wt.} = \frac{(W_3 - W_1) \times 100 \times 100}{(W_2 - W_1) \times (100 - M)}$$

Where, M = Moisture of the sample.

3.4.6 Results

Ash insoluble in dilute HCl = % by weight on dry basis.

3.5 EXPERIMENT 3d: DETERMINATION OF ALKALINITY OF ASH

3.5.1 Principle

Dissolving the total ash in a known volume of standard dilute HCl and titrating the excess acid with standard NaOH determine alkalinity of ash.

Preparation of Sample

Use the ash of the third dish obtained from Experiment 3a.

3.5.2 Requirements

Apparatus

Burette, 10 ml cap.
Conical flask, 250 ml

Chemicals and Reagents

Methyl orange, 0.1% in water
Hydrochloric acid – 0.1N
Sodium hydroxide – 0.1N

3.5.3 Procedure

To the ash in the third silica dish from Experiment 3a, add 10 ml of 0.1 N HCl. Dissolve by warming on a water bath, cool and titrate the excess acid with 0.1 N NaOH using methyl orange indicator. Alkalinity of ash is calculated as potassium carbonate (K_2CO_3).

3.5.4 Observations

Weight of empty dish No. 3	= W ₁	= ---- g
Weight of dish with sample	= W ₂	= ---- g
Weight of dish with ash	= W ₃	= ---- g
Volume of 0.1 N HCl added to the ash	= A	= ---- ml
Titre value (ml 0.1 N NaOH)	= B	= ----ml
Normality of the acid	= N	

3.5.5 Calculations

One ml of 0.1 N HCl is equal to 0.00691 g of potassium carbonate

Therefore, g of potassium carbonate per g of ash =

$$\frac{\text{ml 0.1 HCl required to neutralize the alkalinity of the ash} \times 0.00691}{\text{Weight of the ash}}$$

$$= \frac{(A - B) \times 0.0061}{(W_3 - W_1)}$$

Alkalinity may also be expressed as number of ml of 0.1 N acid required to neutralize the ash from 100 g of the sample.

Alkalinity of ash from 100 g sample =

$$\frac{\text{ml 0.1 N HCl required}}{\text{Weight of sample}} \times 100 = \frac{(A - B) \times 100}{(W_2 - W_1)}$$

3.5.6 Results

Alkalinity of ash = g of potassium carbonate per g of ash, or ml 0.1 acid required to neutralize ash from 100g sample

3.6 PRECAUTIONS

The general precautions mentioned in the course 'Introduction' and those indicated in the experiments should be followed meticulously.

Use a tongs with long handle for keeping and removing dishes from the muffle furnace.