B.Sc. Third Year V Semester

ANALYTICAL CHEMISTRY

Paper XIII [Applied Analytical Chemistry-I]

Analysis of Food & Food Products

2. Analysis of Wheat Flour

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2. Analysis of Wheat Flour

Introduction

- **Food analysis** is the discipline dealing with the development, application and study of analytical procedures for characterizing the properties of **foods** and their constituents.
- **Wheat flour** is a powder made from the grinding of **wheat** used for human consumption. **Wheat** varieties are called "soft" or "weak" if gluten content is low, and are called "hard" or "strong" if they have high gluten content.

- **Protein:** 13.70 g, **Sugars:** 0.41 g,
- **Energy:** 1,418 kJ (339 kcal), **Fat:** 1.87 g.

- Flour plays an important role in the bakery industry. This flour is obtained from wheat so it is necessary to learn about wheat. Wheat is the most important cereal among all the grains. From quality of wheat we can get quality flour. The quality of wheat depends upon the following conditions.

- Soli
- Quality of seeds
- Climate
- Manure
- Farming Techniques

- Wheat are classified in various methods such as: **1. Type, 2. Color, 3. Hardness.**

Types of Wheat Flour

Flour milling

During milling, different parts of the wheat are used or removed at different stages to create different types of flour.

•**Wheatgerm** – This can be white or brown flour with at least 10% made up of wheatgerm added during the milling process.

•**Brown** – This usually contains about 85% of the original grain. During milling some bran and germ is been removed.

•**Wholemeal** – This is made from the whole wheat grain without any additional ingredients or any parts being removed during the milling process.

Organic –This is made from grain that has been grown to organic standards. Growers and millers must be registered and are subject to regular inspections.

Stoneground – This is wholemeal flour ground in a traditional way between two stones using the whole wheat grain with no additional ingredients or any parts being removed.

•**White** – This usually contains around 75% of the wheat grain. During milling most of the bran and wheatgerm is removed.

Here you seen different types of wheat flour and its parts. If you are looking top class Manufacturers, suppliers and exporters of wheat flour then you can visit at web portal of tradeindia

- Hard flour / strong flour
- Weak flour / soft flour.
- From hard wheat we get hard flour. This type of flour contain 11.2 to 11.8% protein, 0.45 to 0.50% ash, 1.2% fat and 74 to 75% starch. The higher protein found in strong flour indicates a higher level of gluten. This type of flour is mainly used for yeast products.
- From soft wheat we get soft flour. This type of flour contains 8.4 to 8.8% of protein, 0.44 to 0.48 of ash and 76 to 77 % starch. Due to the less protein content the flour is mainly used in low structured product like biscuit, cookies, sponges, short and sweet pastes.

Composition of Wheat Flour

STARCH	70%
MOISTURE	14%
PROTEIN	11.5%
ASH	0.4%
SUGAR	1%
FAT OR LIPID	1%
OTHERS	2.1%
ENZYMES [ALBHA & BETA]	

Total energy content is divided into

Analysis of Wheat Flour

1. Determination of Moisture

- **Moisture content** is commonly required for any **flour** specification sheet, with **13.5** percent ideal for soft **wheat** and **14** percent ideal for hard **wheat**. Tempering. ... Liquid water is added to raise the **moisture level** of the **grain** from **12** percent to **17** percent by package materials or from the humidity.
- If more moisture is in the flour it will reduce the storage of life and will induce insect infestation and it may get fungus and bacteria and also it will reduce the **WAP** of the flour. This will result in less yields during the product

Material: *Wheat flour, Oven, Desicator, chemical balance*.

Method:

1. Weigh accurately dried pan with lid. (Note identifier number on pan and lid.)
2. Place 2–3 g of sample in the pan and weigh accurately.
3. Place in a forced draft oven at 130°C for 1 h. Be sure metal covers are ajar, to allow water loss.
4. Remove from oven, realign covers to close, cool, and store in desiccator until samples are weighed.
5. Calculate percentage moisture (wt/wt) as described below.

Calculation

Calculate percentage moisture (wt/wt):

$$\%\ \text{moisture} = \frac{\text{wt of } H_2O \text{ in sample}}{\text{wt of wet sample}} \times 100$$

$$\%\ \text{moisture} = \frac{\left(\begin{matrix}\text{wt of wet sample}\\ +\text{pan}\end{matrix}\right) - \left(\begin{matrix}\text{wt of dried sample}\\ +\text{pan}\end{matrix}\right)}{(\text{wt of wet sample} + \text{pan}) - (\text{wt of pan})} \times 100$$

2. Determination of Ash

❑ The ash content of wheat varies from about **1.50** to about **2.00%**. The pure endosperm contains about **0.35%** ash. Considering that the wheat kernel contains about **80%** endosperm, it becomes clear that the non-endosperm parts of the kernel (pericarp, aleuronic, and germ) are very high in ash when compared to the endosperm.

What is ash in flour?

❑ Ash is the mineral or inorganic material in flour. The ash content of any flour is affected primarily by the ash content of the wheat from which it was milled, and its milling extraction rate: the amount of flour obtained from wheat after milling, when the bran and germ are removed, leaving the endosperm. The test for determining the ash content involves. Incinerating a known weight of flour under controlled conditions Weighing the inorganic residue. Calculating the percentage of ash based upon the original sample weight. The ash value is corrected to dry or other moisture basis for comparison.

Material: *Wheat flour, Desicator, Chemical balance, Muffle furnace, Silica crucible*.

Method:

❑ Weighing a sample of flour or ground wheat (3–5 g) and placing it into an ash cup.

❑ Heating the sample at 585°C (1,085°F) in an electric muffle furnace/oven until its weight is stable (stops decreasing). This process may take several hours.

❑ Cooling residue to room temperature in an desiccator, Weighing residue

❑ Calculating ash content in wheat flour as usual method.

$$\%\ Ash = \frac{Wt.of\ residue\ (g)}{Initial\ Wt.of\ sample\ (g)}\ X\ 100$$

3. Determination of Fat

Introduction:

- Fat should not be more than **1%** in flour.
- It contains coloring pigment carotene which gives color to the flour. There is a higher quantity of oil or fat in the low grade flour then in the high grades.
- The fat or oil when separated from the flour is pale yellowish liquid without taste or smell. Crude **fat contents** were *0.09~1.37%* in the **wheat flour** and *0.07~1.36%* in the dry noodle samples. Based on these results, an acid hydrolysis methods should be used to determine accurate crude **fat contents in wheat flour** and dry noodles.

Material:

1: *5 Sulphuric acid (200 ml concentrated sulphuric acid made unto 1 liter.*
2. *15% solution of sodium tungstate*
3. *Fehling's Solution A: Weigh accurately 69.28 g of copper sulphate and make up the volume to 1 liter with distilled water and filter.*
4. *Fehling's Solution B: Weigh accurately 346g of sodium potassium tartrate and 106g of sodium hydroxide pellets and make up the volume to 1 liter with distilled water. The solution is kept overnight and filtered through glass wood.*
5. *Methylene blue: 1% solution in distilled water.*

Method:

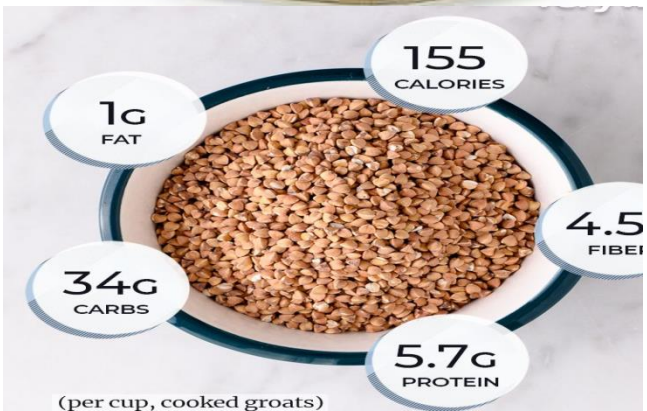
- Place 15g of flour in a 250 ml dry bottle and add 15 ml of water at 27°C. Keep the bottle with the contents at 27°C for one hour, the contents of the bottle being mixed by shaking once every 15 minutes during this time.

- At the end of the digestion period, add 1.5 ml of 1: 5 H_2SO_4 (Reagent No. 1) and 3.5 ml of sodium tungstate (Reagent No. 2) to stop the reaction. Filter immediately through No 1 filter paper and the clear filtrate is used for the determination of sugar content. Take Fehling's solution A (5 ml) and Fehling's solution B (6 ml) in a 250ml conical flask. Place the flour extract in a 50ml burette. Heat the mixed Fehling's solution on a burner and run at least 15-20ml of flour extract into the flask.
- Add 5 drops of methylene blue, heat to boiling and continue boiling for one minute, then add additional extract slowly at a time while still boiling until the blue colour disappears. An extra drop of indicator is helpful at the end. Repeat the titration.
- Calculate % of fat as usual method

Calculation:

$$Percentage\ of\ Fat = \frac{(A - B)}{W} X\ 100$$

Where A = Burette reading of wheat flour sample
B = Burette reading of blank sample
W = Weight of wheat flour taken



Introduction:

4. Determination of Protein

- Flour contains soluble and insoluble proteins. Flour protein consists of
  - Albumin
  - Globulin
  - Gliadin
  - Glutenin
  - Soluble proteins are useful in providing nourishment to yeast during fermentation process for its growth and reproduction. The insoluble protein form a rubbery material when water is added with flour, so when it is mixed and kneaded well, a rubbery material is developed. This is called as gluten. It gives structure to the baked products. Gliadin give extensibility and glutenin gives strength and it holds the gas during baking operation.

- The quality of the flour is decided by the gluten content. If gluten content is more in flour then it is suitable for high structured products like bread.
- This bread making flour should have the gluten from 10 to 11.5%.
- If the flour contains less gluten, then the flour is suitable for lower structured product like cake and biscuits.
- This flour requires low gluten content that is 7 to 10%.

Material:

Kjeldahl digestion flask, HgO, K_2SO_4, anhydrous Na_2SO_4, H_2SO_4, NaOH, Distilled Water, Ammonia & Wheat flour.

Method:

1. Place 1g sample in digestion flask. Add 0.7g HgO or 0.65g metallic Hg, 15g powdered K_2SO_4 or anhydrous Na_2SO_4, and 25 ml H_2SO_4.
2. Place flask in inclined position and heat gently until frothing ceases. If necessary, add small amount of paraffin to reduce frothing. Boil until solution becomes clear.
3. Cool to 25°C and add 200ml distilled water. Then add 25 ml of sulfide or thiosulfate solution and mix to precipitate Hg. Also add few Zn granules to avoid bumping, tilt flask and add NaOH without agitation.
4. Immediately connect flask to distilling bulb on condenser and with tip of condenser immersed in standard acid and 5-7 drops indicator in receiver. Rotate flask to mix contents, then heat until all NH3 had distilled.
5. Remove receiver, wash tip of condenser and titrate excess standard acid in distillate with standard NaOH solution. Correct for blank determination on reagent.

Calculation:

- **% Nitrogen (N) =** [(ml standard acid normality acid) – (ml standard NaOH normality NaOH)] × 1.4007/g sample Multiple % N by 5.7 to get % protein.

Introduction:

5. Determination of Starch

- Starch is not soluble in water until starch is heated to about 140 F with six times of its weight of water. Then the starch cells will swell and the cell wall will burst. Now the starch becomes soluble in water and this process is called as gelatinisation. Starch act as a filler, as it gives rigidity to bread dough. Starch combines with lipid and gluten to retain the gas during fermentation. During milling 6% amount of starch cells are crushed and damaged due to the rollers or type of the wheat or moisture etc. water absorption power of the flour mainly depends upon the damaged starch.
- The damaged starch should not be more than 7 to 9% for bread making. The damaged starch is not essential for cake or biscuit making.
- Hot bread directly from the oven cannot be sliced immediately because the starch is not sufficiently stable and must be allowed to slightly harden. When the bread cools down starch cells shrink and becomes rigid so that the bread can be sliced easily.

❑ Wheat flour contains about 70-80% starch and it is the largest component of flour. Damaged starch is one, which has been physically damaged during the milling process.

Apparatus:

1. Constant temperature water bath regulated at 30° + 1°C. 12
2. Micro burette 10ml capacity
3. Pyrex test tubes 25×200mm
4. Boiling water bath and holder for large test tube.

Reagents:

1. Acetate buffer: Dilute 4.g anhydrous sodium acetate and 3.0 ml glacial acetic acid to1 liter with water and adjust pH to 4.6-4.8.
2. Sulfuric acid solution: Add 100ml reagent grade concentrated sulfuric acid to approximately 700ml water; dilute to 1 liter. Final solution should be 3.68N + 0.05.
3. Sodium tungstate solution: Dissolve 18.0g sodium tungstate in water and dilute to 100ml.
4. Alpha-amylase solution: Dissolve a suitable fungal alpha-amylase preparation (containing 5000 SKB units/g) in reagent No. 1 in proportion of 1.0g enzyme preparation per 450ml buffer. Filter rapidly using three course filter paper. This solution should be used with in 2 hr.

Method:

1. Bring reagent 4 to 30°C. Weigh 1.0g of flour into 100ml stopped conical flask and add 45 ml of reagent 4. Keep it in water bath at 30°C for exactly 15 minutes.
2. At the end of 15 minutes, add 3.0ml of reagent 2 and 2.0ml of reagent 3. Mix thoroughly, let it stand for 2 minutes and filter through Whatman No. 4 filter paper, discarding first 8-10 drops of filtrate.

3. Immediately pipette 5.0ml of filtrate into 25×200mm Pyrex test tube having 10ml of pot ferric acids solution. Immerse test tubes into vigorously boiling water for 20 minutes.
4. Cool test tubes contents under running tap water and pour at once into 100 or 125ml conical flask. Rinse the test tube with 25 ml of acetic acid salt solution. Add 1 ml of soluble starch-KI solution. Mix thoroughly and titrate with 0.IN sodium thiosulfate to complete disappearance of blue colour. Run a blank without sample.

Calculation:

1. Subtract mg maltose equivalent found from Blank-sample.
2. Result of calculations multiplied by 0.092 equals % damaged starch.

% Damaged starch = mg maltose equivalent (B-S) × 0.082. from table.
B = ml of thiosulphate used for Blank
S = ml of thiosulphate used for sample.

6. Determination of Crude Fibers

Introduction:

❑ The dietary fibers is edible parts of plants' carbohydrates that are resistant to digestion in human small intestine. Diets naturally rich in dietary fibers support to prevent constipation, improve gastrointestinal health, glucose tolerance and the insulin response, and reduce the risk of colon cancer, hyperlipidemia, hypertension and other coronary heart disease risk factors. About 45% of the dietary fiber intake comes from grains and grain mixtures

❑ The **fibers content** of whole-**grain wheat** is 12–15% of the dry weight. As they're concentrated in the bran, **fibers** are removed during the milling process and largely absent from refined **flour**. The main **fiber** in **wheat** bran is arabinoxylan (70%), which is a type of hemicelluloses.

Apparatus

1. Wide mouth conical Flask - 1000 ml and 500 ml capacity.
2. Beakers - 500 ml capacity.
3. Fine linen cloth - about 18 threads in a centimeter.
4. Gooch crucible with asbestos.
5. Hot plate.
6. Air - oven (electric).

Reagents required

1. Dilute sulphuric acid 1.25% (W/V).
2. Sodium hydroxide solution 1.25% (W/V).
3. Ethyl alcohol 95% by volume.
4. Ether

Procedure

1. Transfer the material to a one-litre flask and add ether to remove fat, and decant the ether.

2. Take 200 mL of 1.25% (W/V) H_2SO_4 in a beaker and boil it.
3. Transfer the boiling acid to the flask containing the fat-free material and immediately connect the flask with a reflux condenser and heat it to boil the contents for 30 minutes.
4. Stop heating and remove the flask, filter the contents through linen cloth.
5. Wash the residue with boiling water until the washings are free from acid (Check with blue litmus paper).
6. Bring same quantity of sodium hydroxide solution to the boiling under a reflux condenser.
7. Wash the residue through the filter paper, with 200 mL of sodium hydroxide solution.
8. Immediately connect the flask with the reflux condenser and boil for 30 minutes.
9. Remove the flask and immediately filter through the linen cloth.
10. Thoroughly wash the residue with boiling water and transfer into a Gooch crucible, prepared with a thin but compact layer of ignited asbestos.
11. Wash the residue first with boiling water and then with 15 mL of ehtyl alcohol and three successive washings with 15 mL of ether.
12. Dry the Gooch crucible content at 105°C in air-oven for 3 hours, cool and weigh.
13. Repeat the process of drying and weighing until the difference between two consecutive weighings is less than 1 mg.
14. Incinerate the contents of Gooch crucible in an electric muffle furnace at 550°C until all carbonaceous matter is burnt.
15. Cool the Gooch crucible in a desiccator and weigh the ash.

Calculation: $\underset{\text{(\%by weight)}}{\text{Crude fibre (on dry basis)}} = \frac{(W_1 - W_2)}{W} \times 100$

Where,

W = Weight (in gm) of the material taken for the test.

W_1 = Weight (in gm) of the Gooch crucible and contents before ashing.

W_2 = Weight of the Gooch crucible containing asbestos and ash (in gm.)

Note: Any variation in the value of crude fibre from the standard (*i.e.*, 15-30%) norm indicates contamination in the sample.

7. Determination of Acidity

Introduction:

- **Wheat flour** type 750, type 1400 and type 1850 with ash content above 0.60% d.b. were characterized by higher fat **acidity**, in the range of 33-127 mg KOH/100 g d.b. Fat **acidity** in rye **flour** type 500, type 720, type 1400 and type 2000 (in the range of 34-136 mg KOH/100 g d.b.) was higher than in **wheat flours**.


Titrable Acidity

1. **Reagents:** (a) Neutral Ethyl alcohol – 90% (v/ v)
   (b) Standard Sodium Hydroxide solution – approx 0.05 N
   (c) Phenolphthalein Indicator – Dissolve 0.1 gm in 100 ml of 60 % Ethyl alcohol. CEREALS AND CEREAL PRODUCTS.
2. **Procedure:** Weigh 5 gm of sample in a stopper conical flask and add 50 ml of neutral ethyl alcohol. Stopper, swirl gently and allow to stand for 24 hours with occasional swirling. Filter the alcoholic extract through a dry filter paper. Titrate10 ml of the alcoholic extract with standard sodium hydroxide solution to a pink end point using phenolphthalein as indicator Whatman filter paper No.1 is to be used for filtration process. Calculate the % of alcoholic acidity of cereals.

Calculation

$$\text{Alcoholic Acidity with 90\%} = \frac{A \times N \times \text{Factor}\,(0.002452)}{M}$$

……SUGGESTIONS ?

Where, A = Vol. of NaOH used in titration
N = Normality of std. NaOH solution
M = Mass in gm taken for analysis

Created by, Dr. Subhash Lonkar

Decibel (**dB**) is a logarithmic unit that indicates the ratio of a physical quantity (usually power)

Thank you….