

Experiment :3a OIL SEED ANALYSES

Purpose of the experiment:

It is aimed to determine the properties (moisture, oil, ash, pH, nitrogen etc.) of some industrial crops such as sunflower seeds, soybeans, cotton, canola, hazelnut etc. for quality determination.

Quality control of oilseeds is generally carried out to determine pricing and whether the oil content is suitable for the oil industry or not.

At various stages of vegetable oil production, the physical condition and quality of the oil is determined by performing some physical and chemical analyzes. According to the results obtained, it is decided at which stage of the oil treatment step and the state of the oil in this step and whether or not the process should be terminated accordingly.

1. Theory

The oil seeds contain high level of oil, protein, carbohydrate, mineral and vitamins. For this reason, it is an important source of edible oil and protein for human nutrition. Also, it is an important for the industrial as a raw material. The oil crops use many different purposes. For this reason it is called as a wonder crop of the century. The oils produced from the oil crops are used as a source of energy for the human nutrition. Most of the oils (92.1%) used for human consumption comes from oil crops due to higher production cost and limit supply of animal fats. There are plenty of oil seed crops such as soybean, sunflower, rapeseed, peanut, sesame, safflower palm, olive and coconut for crude oil production. Beside crude oil supply, oil crops have other extra benefits. Soybean and peanut are legume crops and supply nitrogen to the soils by symbiotic nitrogen fixation.

The main industrial uses of oilseed plants are;

- Vegetable oil production
- Feed production
- Green or seed shaped animal food
- Crop plant
- Various food and pharmaceutical industry as raw material or additives
- Biodiesel production

Vegetable oils are chemically known as triglycerides of fatty acids and consist of a combination of three fatty acids and one glycerin molecule.

2. Experimental

2.1. Humidity Determination

This analysis is performed to determine the amount of water contained in the oilseed. In oilseeds, moisture is given as mass percentage. If moisture determination is carried out on cleaned seeds, it should be stated as “moisture in clean seed”. The analysis is performed on ground oil seeds. However, milling process is not performed in small grain oil seeds containing dried and semi-dried oil.

Device and Materials:

- General Laboratory Materials
- Sample container
- Oven, desiccator

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Experiment procedure

The oven is pre-operated and heated to 105 °C. Place the sample container in the oven and allow to stand for half an hour. It is then weighed to room temperature in the desiccator and weighed precisely (m_1). The sample of oilseed is crushed in mortar. A portion of 5-10 grams diluted is carefully weighed (m_2) into the sample cup. Place the weighed dish in the oven and dry at a temperature of 105 °C until it reaches a constant scale. The dried sample is weighed in the same container while cooling in the desiccator (m_3).

2.2. Fat Determination

The amount of oil in oil seeds is the total amount of oil extracted from the seeds and is expressed as a percentage. This analysis is carried out by removing all impurities from oil seeds. When expressing the result, it should be noted that the analysis was carried out in pure seed

Device and Materials:

- General Laboratory Materials
- The soxhlet device is shown in Figure 1.
- Hexane or Petroleum ether, Boiling point below 60 °C
- Batch distillation system

Experimental Procedure

The bubble of the soxhlet device is dried in the oven at 105 °C and brought to a constant weight and weighed precisely after cooling in the desiccator (m_1). Approximately 5-10 g of the ground sample is carefully weighed (m_2) cartouche. It is put into the bubble of the soxhlet device. After the addition 3/4 of the volume of petroleum ether into the weighed flask the experimental setup shown in Figure 1 is set up and the medium temperature burner flame or mantle heater is started to be heated. Extraction is carried out for 4-5 hours to be siphoned every 10 minutes. At the end of the extraction, the petroleum ether is distilled off and only the oil remains in the balloon. The balloon is put into a constant weighing at 105 °C. After cooling in desiccator, it is weighed precisely (m_3).

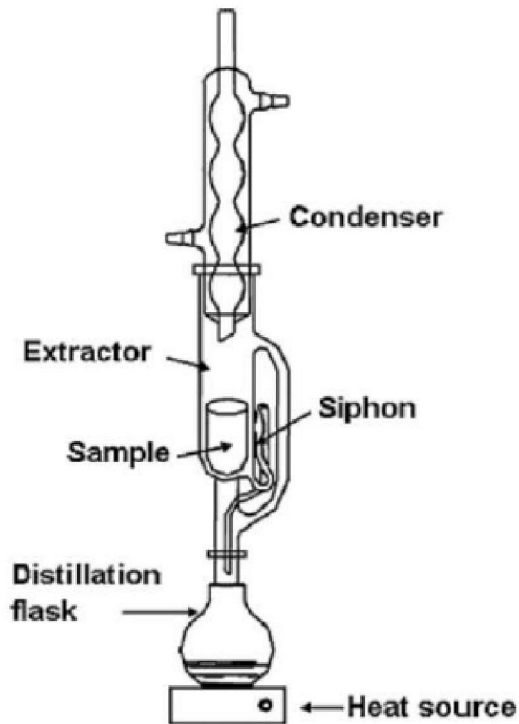


Figure 1. Experimental setup

2.3. Determination of Ash

Device and Materials:

- General Laboratory Materials
- Platinum or Nickel Crucible
- Oxygenated Water

Experimental Procedure:

It is placed in the oven at 700 °C and is weighed in a constant scale. The sample is weighed again by placing approximately 3 g of sample into the crucible (m_2). The sample in the crucible is carbonized in the burner flame. Then the ash is put into the oven at 700 °C. If unburned carbon residues appear in the crucible removed from the furnace, a few drops are wetted with oxygenated water and then put back into the oven and the burning process is completed. At the end of the combustion, the crucible removed from the oven is cooled in the desiccator and weighed precisely (m_3).

2.4. pH Determination

Device and Materials:

- General Laboratory Materials
- Soxhlet Device, seen in Figure 1
- pH Meter
- Petroleum Ether
- Distillable water

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Experimental Procedure:

2 g of the sample is carefully weighed and placed in the soxhlet device for 4 hours with petroleum ether. At the end of the extraction, which is carried out every 10 minutes, the sample is placed on the glass and dried at room temperature. 100 ml of distilled water is added to dry sample in the beaker and placed at $20 \pm 20^\circ \text{C}$ for 1 day. The clear solution above the sample is taken to measure the pH.

2.5. Quantity of Soluble Matter in Water

Device and Materials:

- General Laboratory Materials
- Sохhlet device, seen in Figure 1
- Flask, With Cover
- Grinding device, capable of 50-60 cycles per minute
- Petroleum Ether
- Sulphuric Acid
- Deionized water

Experimental Procedure:

Approximately 10 g of the sample is carefully weighed (m1) and placed in the cartridge and placed in the soxhlet device. After extraction with petroleum ether, the sample taken to the filter paper is left in the open air and the solvent is allowed to fly. Add 500 ml of distilled water at $25-26^\circ \text{C}$ to the sample placed on the lid. The flask with the lid closed is placed in the shaker and shakes for 2 hours at this temperature.

Porcelain capsule is put into constant weighing in 100°C and weighed after cooling in desiccator (m2). At the end of the rinse, the solution in the solution is filtered immediately. 50ml with pipette. the filter is transferred to the porcelain capsule. The capsule is heated in a water bath and the solution is evaporated to dryness. The capsule is put into a constant weighing at 100°C and weighed after drying in a desiccator (m3).

2.6. Determination of Nitrogen

Device and Materials:

- General Laboratory Materials
- Kjeldahl Balloons
- Cooler
- Sulphuric Acid
- The catalyst is composed of a mixture of 100 mg $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ and 1g K_2SO_4 and is used to accelerate disintegration.
- phenolphthalein Solution, 1% (m / m) solution prepared with 95% (m / m) ethanol
- NaOH solution, 40% (m / m)
- Indicator, 0.1% (m / m) Congo red solution or 1 + 5 methyl red + bromokrezol green mixture prepared with 95% ethanol solution of 0.1%
- HCl solution, 0.1 N
- NaOH solution, 0.1 N adjusted

Experimental Procedure:

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Approximately 1g of the sample is weighed precisely (m). If the oil in the sample is more than 5% by weight, the sample is removed by keeping it in the petroleum ether, and the solvent is removed and air dried. This process is not required if the oil percentage is not greater. 20 ml of sulphuric acid is added to the oil-free sample placed on the Kjeldahl flask. A funnel is placed to prevent splashing into the inclined balloon's mouth and the balloon is heated in the small flame. The heating is continued until the skin is completely crushed without boiling the acid. Then about 8gr. catalyst is added. Heating is continued until the solution is clear and almost colorless with a stronger flame. The heated balloon itself is allowed to cool down. 250 ml of water is slowly added to the cooling solution. After placing 1 ml phenolphthalein solution and several boiling stones, they are placed in place in the assembly (Figure 2). 500 ml of flask 100 ml of 0.1 N HCl solution is placed and the outlet end of the cooler is immersed in the acid. The solution in the flask is made basic by dropping 40% NaOH solution into the separation funnel. The fact that the solution is basic means that the color turns red. The solution in the flask is heated and heated. Ammonia released by the reaction is absorbed by the acid solution. After the ammonia is removed from the cooler, the tip of the cooler is washed with distilled water and added to the solution. A few drops of indicator are added to the solution and the excess acid is titrated with the adjusted NaOH solution. The volume V_1 of the spent base is recorded.

The process performed so far is repeated without sample preparation and the volume (V_2) of the base spent in the witness test is determined.

3. Calculations

The expressions of the values that should be calculated in each analysis of the measurements are summarized below.



The mass percentage (R) of the moisture in the sample is calculated from Equation 1.

$$R = \left[\frac{m_2 - m_3}{m_2 - m_1} \right] \times 100 \quad (1)$$

Here;

m_1 : Mass of the container, gr

m_2 : Sample + Container mass, gr

m_3 : Dried sample + Container mass, gr

❖ The mass percentage (Y) of the oil in the sample is calculated from Equation 2.

$$\%Y = \left[\frac{m_3 - m_1}{m_2} \right] \times 100 \quad (2)$$

Here;

m_1 : The mass of the balloon, gr

m_2 : The mass of the sample, gr

m_3 : Oil + Balloon mass, gr

❖ The mass percentage (K) of the ash in the sample is calculated from Equation 3.

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$$\%K = \left[\frac{m_3 - m_1}{m_2 - m_1} \right] \times 100 \quad (3)$$

Here;

m_1 : The mass of crucible, gr

m_2 : Specimen + crucible mass, gr

m_3 : Ash + crucible mass, gr

- ❖ The pH reading from the device is given in decimal.
- ❖ The mass percentage (S) of the water-soluble substance in the sample is calculated from Equation 4.

$$\%S = \left[\frac{m_3 - m_2}{m_1} \right] \times 1000 \quad (4)$$

Here;

m_1 : Sample quantity, gr

m_2 : The mass of crozen, gr

m_3 : Residue + Croze mass, gr

- ❖ The mass percentage of the sample nitrogen (A) is calculated from Equation 5.

$$A = \left[\frac{1,4 \times N \times (V_2 - V_1)}{m} \right] \quad (5)$$

Here:

N: concentration of adjusted NaOH solution, N

V_1 : Volume of base spent in experiment, ml

V_2 : The volume of the base spent in the experiment, ml

m: mass of sample, gr

4. Experimental Results

At least one of the assay tests given in Section 2 is carried out in the course of laboratory work (preferably fat determination) and the results are presented in a table format given below, in comparison with the required values.

Table 1. Experimental Results

	Experimental result	Standard Value	Deviation
%Moisture			
% Fat			
% Ash			
pH			
%Soluble matter in water			
% Nitrogen			

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QUESTIONS

1. Explainthechemical structure of oilseeds.
2. What is triglyceride and glycerin? What type of reaction they give?
3. What is the production steps of vegetable oil production process?
4. What additional nutrients do they contain when used as a nutrient?
5. What is extraction? What is the extraction types?
6. How can we make material balance in solid-liquid extraction systems?
7. According to the related experiment derive an equation for soluble matter in water?
8. In the nitrogen determination experiment what is the reason of making basic solution? Can ammonia be used instead of NaOH?
9. Taking into account the relevant experiment,derive an equation giving the amount of nitrogen in the oilseed.