KEMENTERIAN PENGAJIAN TINGGI
JABATAN PENDIDIKAN POLITEKNIK DAN KOLEJ KOMUNITI

POLITEKNIK MALAYSIA
TUN SYED NASIR

OIL AND FAT PROCESSING LABORATORY

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Oil and Fat Processing Laboratory

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Appreciation

Assalamualaikum W.B. T

Be grateful to Allah S.W.T for his abundance and his secret to us in preparing this oil and fat processing laboratory module.

In this regard, we look at both our parents and our families as we always wish for our success in whatever we do.

We also wish to thank the Director, Deputy Director, department heads, and all our colleagues at Tun Syed Nasir Syed Ismail Polytechnic (PTSN) and who always provide guidance to us. We will remember this friendship anytime.

The most special tribute to our students at PTSN is our main model for achieving this mission.

We hope that this module will help the lecturers who teach this course to do their job better. Hopefully, this module will also help students better understanding and guided in oil and fat processing for achieving learning objectives.

Finally, thank you to everyone involved in helping us prepare this module. All your services are greatly appreciated.

Preface

Oil and fat processing laboratory covers the study of various stages and methods employed in oil and fat processing., beginning storage, extraction of crude oil from animal and plant sources to refining and modification of extracted oils and fats. Raw materials for oil and fat extraction include animal by-products, fleshy fruits and oilseeds. Crudes oils and fats from these sources can be recovered by methods which include mechanical extraction, thermal extraction, solvent extraction and enzymatic hydrolysis. Some oils, such as virgin olive oil, are ready for consumption after this initial step (pressing), while others require additional processing such as refining and modification. The extent of oil and fat processing depends n the source, quality and nature of the end use.

Content

POLITEKNIK MALAYSIA
TUN SYED NASIR

LABORATORY REPORT COVER PAGE

Title of experiment:			
Group number:		**Program/Semester:**	
Date of experiment:		**Date of submission:**	
Name & Reg. No:	**1.**		
	2.		

Criteria	Marks Weight (%)	Competent	Towards Competency	Satisfactory Performance	Towards Satisfactory	Unsatisfactory Performance	Marks
		5	4	3	2	1	
A. Procedures And Techniques	5	Excellent technique was used throughout the lab procedure.Procedures were well-planned and well-executed.	No errors in technique were observed during the lab procedure.Procedures were well-planned and were carried out in an organized fashion.	Only minor errors in technique were observed during the lab procedure. Procedures were carried out well but may have been slightly disorganized.	Only a few errors in technique were observed during the lab procedure, but they may have been significant. Procedures may not have been well-planned, or they may have been carried out in a disorganized fashion.	Several serious errors in technique were observed during the lab procedure. Procedures were not well-planned and were carried out in a disorganized fashion.	
B. Data Collection And Observations	5	Data and observations were recorded accurately, descriptively, and completely, with no serious errors.	Data and observations were recorded accurately, descriptively, and completely, with only minor errors.	Data and observations were recorded accurately, with only minor errors or omissions.	Data and observations were recorded adequately, with some errors or omissions.	Most data and observations were recorded adequately, but with several significant errors or omissions.	
C. Calculations, Data Analysis Or Tabulation	5	Calculations and data analyses were performed clearly, concisely, and accurately, with correct units. Graphs or diagram, if necessary, were drawn accurately and neatly and were clearly labeled.	Calculations and data analyses were performed accurately, with correct units and properly worked-out calculations, but the work may have been slightly unclear or disorganized. Graphs or diagram, if necessary, were drawn accurately and neatly.	Calculations and data analysis were performed accurately, but some minor errors were made either in calculations or in applying correct units. Graphs or diagram, if necessary, were drawn accurately and neatly.	Calculations and data analysis were performed accurately, but minor errors were made both in calculations and in applying correct units. Graphs or diagram, if necessary, were drawn adequately.	Calculations and data analysis were performed inaccurately, but correct units were used most of the time. Graphs or diagram, if necessary, were drawn adequately.	
D. Discussion	15	Students recognized the connections between their observations and the related concepts in fat and oil processing; this understanding was expressed clearly and completely in the report.	Students effectively expressed their recognition of the connections between their observations and the related concepts in fat and oil processing. Reasoning was good in the report.	Students satisfactorily expressed their recognition of the connections between their observations and the related concepts in fat and oil processing. Reasoning was occasionally weak in the report, but only in a few places.	Students recognized connection between their observations and the related concepts in fat and oil processing, but this understanding was very weakly expressed in the report. Reasoning was generally weak throughout the report.	Students may not have recognized connections between their observations and the related concepts in fat and oil processing; no expression of understanding was evident in the report.	
E. Conclusion	10	All conclusions are written with full attention to task/laboratory objective.	Almost all conclusions are written with full attention to task/laboratory objective.	Conclusion is generally written with attention to task/laboratory objective.	Partially conclusion were concluded .	Conclusion witten out of the task/laboratory objective.	
F. Questions	5	Answers to questions were complete and were written correctly and accurately.	Answers to questions were written correctly and accurately but may have revealed minor misunderstandings.	Answers to most questions were correct, but there are some misunderstanding or minor errors.	Some answers to questions were incorrect because of misunderstandings, minor errors, or poor data.	Some answers to questions were incorrect or poorly written.	
G. References	2.5	All reputable background sources were used, quoted correctly and attached together.	Almost all reputable background sources were used, quoted correctly and attached together.	A few reputable background sources are used and quoted correctly. Information is translated using student's own words.	Most reputable background sources are used, quoted incorrectly and no attachment.	Unreputable background sources are used, quoted incorrectly and no attachment.	
H. Formatting	2.5	All particulars are clearly written according to the task/laboratory sheet.	Almost all particulars are clearly written according to the task/laboratory sheet.	Some particular are clearly written according to the task/laboratory sheet.	Most particulars are incomplete.	Few particulars are incomplete.	
TOTAL MARK							

LABORATORY SAFETY RULES

Lab Safety Rules

Science labs offer great opportunities for learning, teaching, and research. They also pose hazards that require proper safety precautions.

Dress appropriately

Tie back long hair, and wear suitable gloves, goggles, and other protective equipment.

Proper supervision

Don't perform lab experiments without instructor supervision (unless given permission to do so).

Know location of emergency numbers & safety equipment

Know the location of safety equipment and emergency phone numbers (such as poison control) so you can access them quickly if necessary.

No food

Don't eat or drink in the lab—and never taste chemicals.

ID hazards

Identify hazardous materials before beginning labs.

Be attentive

Be attentive while in the lab. Don't leave lit Bunsen burners unattended or leave an experiment in progress.

Be careful when handling hot glassware

Turn off all heating appliances when not in use. Keep flammable objects away from your workspace.

Keep a clean workspace

Don't obstruct work areas, floors, or exits. Keep coats, bags, and other personal items stored in designated areas away from the lab. Don't block sink drains with debris.

Handle glassware carefully

Properly dispose of anything that breaks. Report cuts, spills, and broken glass to your instructor immediately.

Clean up

After completing the lab, carefully clean your workspace and the equipment, and wash your hands.

GENERAL GUIDELINES

1. Conduct yourself in a responsible manner at all times in the laboratory.
2. Follow all written and verbal instructions carefully. If you do not understand a direction or part of a procedure, **ASK YOUR INSTRUCTOR BEFORE PROCEEDING WITH THE ACTIVITY**.
3. Never work alone in the laboratory. No student may work in the lab without the presence of the instructor.
4. When first entering the laboratory, **DO NOT TOUCH ANY EQUIPMENT, CHEMICALS, OR OTHER MATERIALS IN THE LABORATORY AREA UNTIL YOU ARE INSTRUCTED TO DO SO**.
5. Perform the experiments as authorized by your instructor. Carefully follow all instructions, both written and oral. Unauthorized experiments are not allowed.
6. **DO NOT EAT , DRINK , OR CHEW GUM IN THE LABORATORY**. Do not use laboratory glasswares as containers for food or beverages.
7. Be prepared for your work in the laboratory. Read all procedures thoroughly before entering the laboratory. Never fool around in the laboratory. **HORSEPLAY, PRACTICAL JOKES, AND PRANKS ARE DANGEROUS AND PROHIBITED.**
8. Always work in a well-ventilated area.
9. Observe good housekeeping practices. **WORK AREAS SHOULD BE KEPT CLEAN AND TIDY AT ALL TIMES.**
10. Be alert and proceed with caution while in the laboratory. Notify the instructor immediately of any unsafe conditions you observe.
11. Dispose of all chemical wastes properly. Never mix chemicals in sink drains. Sinks are to be used only for water. Check with your instructor for disposal of chemicals and solutions.
12. Labels and equipment instructions must be read carefully before use. Set up and use the equipment as directed by your instructor.
13. Keep hands away from face, eyes, mouth and body while using chemicals or lab equipment. Wash your hands with soap and water after performing experiments.
14. Experiments must be personally monitored at all times. Do not wander around the room, distract other students, startle other students or interrupt with the laboratory experiments of others.
15. Be aware of the locations and operating procedures of all safety equipment including: first aid kit(s), and fire extinguisher. Notice where the fire alarm and the exits are located.
16. Be aware of what to do if there is a fire drill during a laboratory period; containers must be closed, and any electrical equipment must be turned off.

CLOTHING

1. At any time, chemicals, heat, or glassware are used, students must wear safety goggles. **NO EXCEPTION TO THIS RULE!**
2. Contact lenses must not being worn in the laboratory.

3. Dress properly during a laboratory activity. Long hair, dangling jewelry, and loose or baggy clothing are a hazard in the laboratory. Long hair must be tied back, and dangling jewelry and baggy clothing must be secured. Shoes must completely cover the foot. No sandals are allowed in the laboratory.
4. A lab coat or smock should be worn during laboratory experiments.

ACCIDENTS AND INJURIES

1. Report any accident (spill, breakage, etc.) or injury (cut, burn, etc.) to the instructor immediately, no matter how trivial it may seem. Do not panic.
2. If you or your lab partner is hurt, immediately (and loudly) yell out the instructor's name to get the instructor's attention. Do not panic.
3. If a chemical splash in your eye(s) or on your skin, immediately flush with running water for at least 20 minutes. Immediately (and loudly) yell out the instructor's name to get the instructor's attention.

CHEMICAL HANDLING

1. All chemicals in the laboratory are to be considered dangerous. Avoid handling chemicals with fingers. Always use a tweezer. When making an observation, keep at least 1 foot away from the specimen. **DO NOT TASTE, OR SMELL ANY CHEMICALS.**
2. Check the label on all chemical bottles twice before removing any of the contents. Take only as much chemical as needed.
3. **NEVER** return unused chemicals to their original container.
4. **NEVER** remove chemicals or other materials from the laboratory area.

GLASSWARE AND EQUIPMENT HANDLING

1. Never handle broken glass with your bare hands. Use a brush and a dustpan to clean up broken glass. Place broken glass in the designated glass disposal container.
2. Examine glassware before each use. Never use chipped, cracked, or dirty glassware.
3. If you do not understand how to use a piece of equipment, **ASK THE INSTRUCTOR FOR HELP!**
4. Do not immerse hot glassware in cold water. The glassware may shatter.

EXPERIMENT 1: OIL AND FAT EXTRACTION – WET RENDERING

Objectives:

i. To carry out the animal fat rendering process by using the wet rendering method.
ii. To determine the yield of fat produced.
iii. To analyze the fat produced.

Introduction

Rendering systems are divided into two classes: edible rendering of animal fatty tissue into edible fats and proteins for human consumption, and inedible rendering of animal by-product materials into animal fats and proteins for animal feed and other non-edible applications. The inedible rendering process consists of two basic steps: "cooking" or moisture removal by evaporation, and separation of the melted fats from the protein solids. The basic rendering process involves the use of batch cookers. In the recent years, continuous rendering systems which utilize continuous cooking have replaced many batch systems.

A continuous system is described for edible rendering. This system features two stages of centrifuges:

1. A horizontal, solid bowl centrifuge for the separation of the protein solids from the liquid.
2. A disc centrifuge for the separation of the edible fat from the sludge phase consisting of protein fines and water.

In the process of rendering, meat scraps are heated in steam or water to cause the fat to melt. The melted fat then rises and water and remaining tissue settled below. The melted fat is then separated by skimming or centrifugation. Dry-heat rendering cooks the tissue in a vacuum to remove moisture. Wet rendering utilizes water and steam, andnd low-temperature rendering uses just enough heat to melt the fat.

Temperatures of between 240° and 295°F (115° to 146°C) is used in the rendering process, which is more than sufficient to kill bacteria, viruses ,and many other microorganisms as to produce an aseptic protein product that is free of potential biohazards and environmental threats.

Material:

Water

Chicken skin

Apparatus:

500ml beaker

Spatula

Hot plate

Filter

Procedure:

1. Weigh 100g of chicken skin in a 500ml beaker.
2. Pour 250ml of water into the beaker. Heat the sample at 115° to 146°C for two hours.
3. After two hours, separate the chicken skin with the stock (water and fat) by using a filter.
4. Cool the stock solution and place it in the chiller until the fat is solidified and formed of two layers.
5. Skim the upper layer of fat to separate it from the water. After the skimming process, melt the fat and proceed with free fatty acid analysis and moisture content.

Analysis:

Discussion

Provide a relevant discussion such as the methods principle, the important of the analysis, observation, data analysis and error that may arise from this experiment.

Conclusion

Provide a relevant conclusion based on this experiment.

Questions

1. What is the best method that may be used to determine very low moisture content in oils (~1% or less)?
2. Suggest other methods tp determine the moisture content in oils.

Reference

Provide the sources of reference for your discussion.

EXPERIMENT 2 : MECHANICAL OIL EXTRACTION – HYDRAULIC OIL PRESS

Objectives:

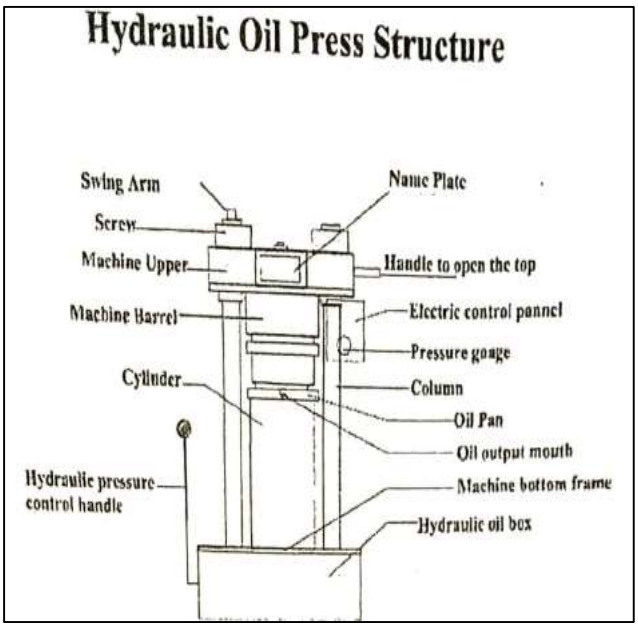
iv. To carry out vegetable oil extraction using a hydraulic press method.

v. To determine the yield of oil produced.

Introduction

Oil extraction by pressing was historically conducted using devices operated by stones and levers to exert direct pressure on the seeds. An improved form of mechanical device, that allows the application of considerably higher pressure on seeds, involves the use of the hydraulically operated press. These devices have evolved from the manual to motorized systems that press the seeds and release the oil and the press cake. The next improvement in oil extraction was the screw press, also called the expeller. This type of press enables continuous operation rather than the batch processing imposed by hydraulic presses.

Full pressing extraction is used for “high oil” seeds (>30%) resulting in a 4.5%–7.5% residual oil content in the meal. This process is no longer widely used because it is difficult to obtain the desired oil yield without damaging the product quality, due to the high pressure and heat generated in the press. It is used only for small-scale operations, and/or for niche and genetically modified organism (GMO) products. It can also be used to produce the so-called “cold-pressed oil,”which is a high-quality product. In this treatment, flakes or even whole seeds are pressed without having to undergo a prior heat treatment or cooking. The residual oil in the cake is around 10%–20% and it can then be recovered by cooking the cake and subjecting it to a second pressing. The oil resulting from the second pressing requires a full refining treatment before becoming edible, whereas clarification and bleaching may be sufficient for the cold-pressed oil. Fines contained in the press oil are removed in two stages. Firstly, the pressed oil is allowed to settle in a tank, and afterward, it is further clarified by means of filters (small plants) or decanters (large plants).



The automatic hydraulic oil press is one of the most advanced equipment and it is the best choice compared to the hand-operated workers. It is the easiest machine for people to operate and replace wearing parts least with the highest oil yielding rate among all range of machines. Besides, it is particularly suitable for people from populated cities to spend less money to buy real goods because of less amount of squeezing per time(2-8 kg/time), and it needs shorter time(8-12 minutes/time) for processing. In addition, it can ensure fast processing is giving materials in rural towns, which is known as assured oil. The main processing oil crops are: olives, cashew nut, sesame, walnut, camellia, pine nuts (Peeling most effective), almond and other high oil crops.

Material:

Choose any oilseeds or oil-bearing fruit: coconut meat, olives, avocado, cashewnuts, sesame, peanut, walnut, camellia, pine nuts (peeling most effective), almond, pumpkin seeds, moringa seeds, and other high oil crops.

Apparatus:

Hydraulic Oil Press, Top Loading Balance (up to 3-4kg range)

Procedure:

1. Preheat the oil press system at a temperature of 65 ℃.

2. Weigh an oil collection container and 1.5 to 2.5kg of oilseeds on a top-loading balance. Refer your instructor on the actual oilseeds amount.

(Never use the analytical balance for this step)

3. Roast the pre-weigh oilseeds on a wok with mild fire.

4. Ensure the wrench handle of the top ejector plate is opened completely. The angle between the top ejector plate and the work position is 180 degrees, and the mat should be put in the bottom of the chamber. Put the piston in place for preparation. The machine without oilseeds should be run 1-2 times before extruding.

5. Insert the roasted oilseeds into a sack and make a knot. Insert the filled sack into a second sack, and make a knot.

6. Raise the chamber platform so it dropped lower by 10mm from the upper cove by running the hydraulic pump. Then fill the oil press chamber with layers of bottom disk (perforated) → mat → oilseed-filled sacks → mat → stainless steel disk. The chamber platform can be lowered by making the manual valve control handle upright.

(*Be careful with the heavy steel disk. Slide them into the chamber carefully)*

7. Close the wrench handle of the top ejector plate completely.

8. Place a pre-weigh oil collection container below the hose.

9. Put the control handle in the downward position. Run the hydraulic pump. The press occurs in three cycles, between 40Mpa and 60Mpa.

(*Never leave the machine unattended while running)*

10. When it is done, raise the control handle in upright position. Open the wrench handle of the top ejector plate. Raise the chamber platform and carefully take out all the disks, mats and pressed cake.

(The chamber is hot, use appropriate PPE)

11. Weigh the oil collected and proceed for moisture content analysis using a Moisture Analyzer.

12. Switch off the oil press and clean parts of the machine.

Result

Mass of roasted oilseeds, g	
Mass of oil collected, g	
Moisture content of extracted oil, %	

Yield Calculation

$$\text{Oil Yield} = \frac{Mass\ of\ Oil\ Collected}{Mass\ of\ Roasted\ Raw\ Material} \times 100\%$$

Discussion

Provide a relevant discussion such as the methods principle, the important of the analysis, observation, data analysis and error that may arise from this experiment.

Question

1. Explain the differences between cold and hot oil press method.

2. Why roasting is a crucial step before oil pressing?

3. Suggest and elaborate a method to extract the remaining oil from the pressed cake.

Conclusion

Provide a relevant conclusion based on this experiment.

Reference

Provide the sources of reference for your discussion.

EXPERIMENT 3: OIL AND FAT EXTRACTION - SOLVENT EXTRACTION

Objectives:

i. To carry out extraction process by using solvent extraction method.
ii. To determine the yield of sample produced.

Introduction.

Fat plays an important role in many kind of food. Fat contributes to the flavour of food as well as it gives texture and also mouthfeel to the food. It is an essential component which gives us maximum energy. Approximately 9 Kcal energy per gram. Excessive intake of fat mostly leads to obesity and its insufficienly leads to malnutrition. It nourishes the body with all the essential fatty acid that body can not synthesise and also help in building the body.

Hence there is the need to measure the amount of fat present in food, so that we will have a better idea of its count and manage our diet accordingly. It also helps in extracting all the oil present in food.

There are two ways to find out the fat present in food, either by acid hydrolysis or by solvent extraction. The solvent extraction method is more pronouncedly known as Soxhlet method and it came into the scene in 1897. This method is widely used in almost all food industries and primarily used in oil extraction industries.

PRINCIPLE – SOXHLET EXTRACTION METHOD

Lipids in food present in various forms like monoglycerides, diglycerides, triglycerides, sterol, free fatty acid, phospholipid and carotenoids as well as fat-soluble vitamins. Lipid is soluble in organic solvent and insoluble in water and because of this, organic solvents like hexane, petroleum ether have the ability to solubilize fat and fat is then extracted from food in combination with the solvent. Later the fat is collected by evaporating the solvent.Almost all solvent is distilled off and can be reused.

SOLVENT PROPERTIES:

Primarily solvents like hexane and petroleum ether are in use due to their low boiling point. In addition , the solvent possesses following properties:

1. Distribution Coefficient: This is the ratio (at equilibrium) of the concentration of solute in the extract and raffinate phases.
2. Selectivity (Separation Factor): If there are more than one solutes, then we have to choose the appropriate solvent due to the chances of intermixing.
3. Insolubility of Solvent: The solvent should have low solubility in the feed solution.
4. Recoverability: The solvent should be thermally stable at the distillation temperature due to its volatility.
5. Density: Density should be lower than water.
6. Interfacial Tension: The larger the interfacial tension, the more difficult the dispersion of one liquid in the other will be.
7. Chemical Reactivity: The solvent should be stable chemically and inert.
8. Non-toxic

Sample preparation:

First of all, we have to dry the product and remove any moisture in order to facilitate the entry of the organic solvent, because moisture restricts the entry. Then the size reduction is there to increase the surface area and due to it, there is a larger exposed surface.

Material:

Ground nut/ any sample

Chemical:

n-hexane/ Petroleum ether (Boiling temperature 60°-80°c)

Apparatus:

Weighing balance

Soxhlet apparatus

Drying oven

Thimble

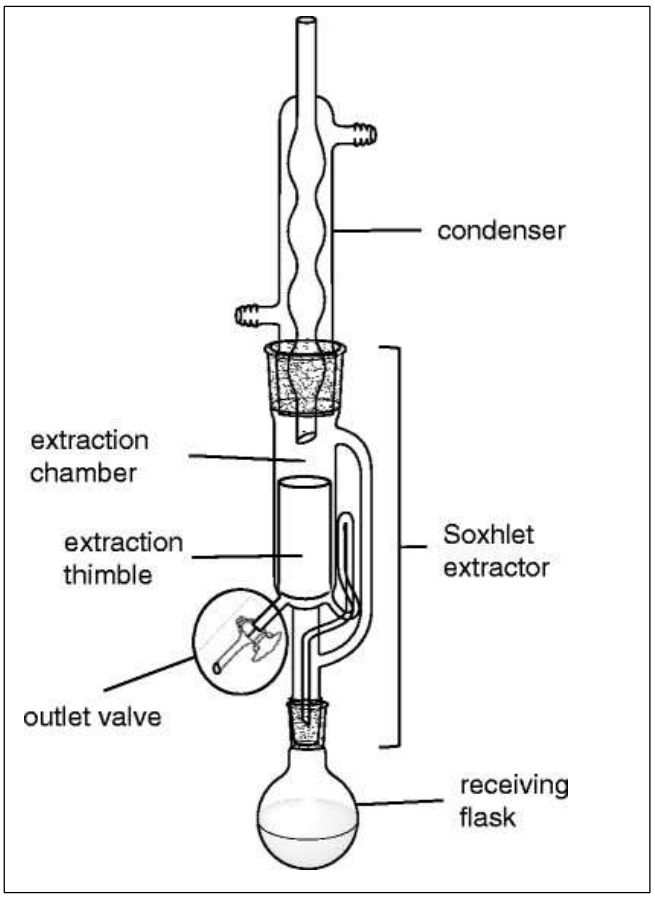
Heating mantle

Glass rod

Desiccator with silica gel

Cotton plugs

Rotary evaporator



Soxhlet extraction diagram

Procedure:

1. First of all, rinse all the glass apparatus with petroleum ether and dry it in the oven at 102°c and remove it before keeping it in the desiccator.
2. Weigh 25 gram of grounded and dried sample and place it in the thimble.
3. Place the thimble in the soxhlet extractor.
4. Take a 250ml round bottom flask clean it and fill the flask with 200 ml of petroleum ether.
5. Place the whole setting on a heating mantle and allow the petroleum ether to boil.
6. Continue the extraction process for several hours, approximately six hours.
7. Remove the condensing unit from extraction unit and allow the sample to cool down.
8. Use rotary evaporator to separate the sample and solvent.
9. Collect all solvent after distillation.
10. Place the sample in the oven and place it in the desiccator after removing it from the oven.
11. Take the weight of the sample.
12. As a result, we get a defat sample.

Result Calculation:

Empty thimble= w1

Thimble with sample= w2

Weight of sample= p

Then crude fat percentage in g= (w2-w1)／p × 100

This method is an efficient method to extract all fat present in food. Hence it is used in oil extraction units for better recovery of oil. This method is also applied to the deoiled cake which is collected from screw impellers rather than high-pressure expression. It is also used in the analysis of fat present in the sample.

Discussion

Provide a relevant discussion such as the methods principle, the important of the analysis, observation, data analysis and error that may arise from this experiment.

Conclusion

Provide a relevant conclusion based on this experiment.

Questions

1. Explain the purpose of solvent in soxhlet extraction.
2. Why rotary evaporator is used?
3. Explain the principle of reflux in soxhlet extraction.
4. Discuss the percentage of yield of sample.

Reference

Provide the sources of reference for your discussion.

EXPERIMENT 4: OIL REFINING -DEGUMMING, NEUTRALIZING, WASHING, AND BLEACHING OF CRUDE PALM OIL

Objectives:

iii. To carry out degumming, neutralization, washing, and bleaching on crude palm oil (CPO) in a batch process

iv. To determine the yield of neutralized palm oil

v. To analyze and compare the quality of crude and processed palm oil.

Introduction:

There are several refining processes for edible oil and the purpose of the processes is to convert the crude oil or fat into a product that is more suitable for its end purpose. This will involve the removal of undesirable components and usually results in a product with minimal color and flavor. The processes have been devised to minimize changes in the triacylglycerols and in the levels of those minor components which confer nutritional benefit. If the oil is to be processed with the aid of a catalyst (hydrogenation, interesterification, etc.), it is also important to remove or minimize poisons that reduce catalyst efficiency. The compounds removed by refining include phospholipids, free acids, mono- and di-acylglycerols, color, trace metals, oxidation products, and environmental

Process	Procedure	Impurities removed or reduced
Degumming	H_3PO_4, H_2O, 70–80°C	Phospholipids, trace metals, carbohydrates, proteins
Neutralisation	NaOH or other alkali	Fatty acids. phospholipids, pigments, trace metals, sulfur compounds
Washing	Water	Soap
Drying	–	Water
Bleaching	bentonite, etc.	Pigments, oxidation products, trace metals, sulfur compounds, phospholipids, traces of soap
Filtration	–	Spent bleaching earth
Deodorisation or physical refining	Steam at reduced pressure	Fatty acids, mono- and di-acylglycerols, oxidation products, pigment decomposition products, pesticides
Polishing	–	Traces of oil insolubles

contaminants. The processes are summarised in the table below. Some of the `impurities' removed in the refining processes are valuable by-products that can be recovered and used.

Oils may be refined by a series of processes that can be grouped as `chemical refining' or `physical refining'. The former involves degumming, chemical neutralization, bleaching, and deodorization, while the latter requires only bleaching and steam distillation (deodorization). Physical refining is preferred as it is a more economical process requiring fewer chemicals, producing less waste, and giving higher oil yields. The advantages are particularly apparent with oils (such as palm oil) in which contains high levels of free acid and low levels of phospholipids. The chemical method is preferred for oils with high phospholipid levels and for cottonseed oil which contains gossypol that can only be conveniently removed by alkali treatment.

The SOLTEQ-QVFTM Oil Neutralizer / Washer / Bleacher (Model: BP515) is a lab-scale batch processing vessel capable of carrying out the neutralization, washing, and bleaching processes on small batches of crude edible oils, as part of the edible oil refining process.

Removal of free fatty acids (FFA) is done by the neutralization process, whereby crude edible oil is neutralized with an alkali solution – caustic soda, forming a water-soluble soap. The soap is subsequently removed by washing, gravity settling, and draining. The oil is dried by it heating in a vacuum to remove any remaining water. Colour and impurities are removed by mixing the oil with an adsorbent – bleaching earth, which is then removed by filtration to leave a clean oil for further processing.

Figure 3.1: Oil Neutraliser / Washer / Bleacher (Model: BP515)

No.	Component	No.	Component
1	Mixer, M1	6	Earth Hopper. T3
2	Mixer Oil Seal Reservoir, T4	7	Control Panel
3	Reagent Tank, T2	8	Batch Reactor, R1
4	Filter, S1	9	Vacuum Pump, L1
5	Feed Tank, T1	10	Filter Pump, P1

Material:

Crude Palm Oil

Phosphoric acid

Sodium hydroxide solution

Distilled water

Bleaching earth

Apparatus:

Measuring Cylinder 10mL

Measuring Cylinder 2L

Glass rod

Spatula

Beaker

Analytical Balance

Operating Procedures:

You may refer to the process flow chart at the end of this manual for a clear picture

A. General Start-up Procedures

1. Ensure that heater W1 and W2 switches are in the off position on the control panel.
2. Power up the control panel with the Main Switch.
3. Ensure that all valves are initially closed.
4. Check and if necessary, adjust the pressure regulator PR01 to 3 kgf/cm2 and PR02 to 2 kgf/cm2
5. The unit is now ready for the experiment.

B. Pre-Refining

1. Perform the general start-up procedures as described in A.
2. Prepare 25 L of CPO in feed tank T1.
3. Fully open HV12 to allow water into the sealant ring of vacuum pump L1. Maintain waterflowrate at 3 LPM.
4. Open HV02 and HV13.
5. Start L1 to create a vacuum 0.3 bar abs (PT01) and draw in the CPO from feed tank T1.
6. Stop the vacuum pump when T1 is empty. Close HV02. Open HV14 to return R1 to atmospheric. Close HV12.

C. Degumming

1. Set the temperature of TIC102 for R1 to 60 °C. Turn on heater W2.
2. Set stirrer speed to 100 rpm and start stirrer M1 for a gentle mixing.
3. Prepare 0.02-0.04% v/v of phosphoric acid using the formula below

$$\frac{x\ ml\ of\ phosphoric\ acid}{25{,}000mL\ of\ crude\ palm\ oil} \times 100 = 0.030\%$$

4. When TIC102 has achieved set temperature, open HV04 to dose the phosphoric acid.
5. Set a timer for 5minutes.

D. Neutralization

1. Set the temperature of TIC102 for R1 to 60 °C. Turn on heater W2.
2. Set stirrer speed to 100 rpm and start stirrer M1 for a gentle mixing to avoid emulsification with water.
3. Prepare 1.5 L of 10% sodium hydroxide in reagent tank T2. Note: Add small amounts of NaOH slowly into the water with mixing as this is an exothermic reaction.

$$\frac{x\ g\ of\ NaOH}{1{,}500mL\ of\ NaOH\ solution} \times 100 = 10\%$$

4. When TIC102 has achieved set temperature, open HV03 to feed into R1.

5. Set a timer for 10 minutes.

6. Stop M1 and allow 1 hour for the soapstock to separate and settle by gravity.

7. Drain the soapstock with HV06 and HV07.

8. Record the volume of soapstock collected.

E. Washing

1. Increase the set temperature of TIC102 for R1 to 80 °C.

2. Set stirrer speed to 100 rpm and start stirrer M1 for a gentle mixing to avoid emulsification with water.

3. Prepare 2 L of distilled water in reagent tank T2.

4. Set the temperature of TIC101 for T2 to 40 °C. Start heater W1 to pre-heat the water.

5. When TIC102 has achieved set temperature, turn off heater W1 for tank T2, then open HV03 to feed into R1.

WARNING: Ensure heater W1 is switched off before draining the reagent tank T2 to prevent overheating

6. Set a timer for 10 minutes.

7. Stop M1 and allow 30 minutes for wash water to separate by gravity.

8. Drain the wash water with HV06 and HV07.

9. Record the volume of water collected.

10. Calculate the volume of Neutralized palm oil that remains in the reactor:

The volume of NPO = Volume of (CPO + H3PO4 + NaOH + Washing water) – Volume of Soapstock (in D) – Volume of water (in E)

F. Bleaching

1. Increase the set temperature of TIC102 for R1 to 90 °C.

2. Set stirrer speed to 200 rpm and start stirrer M1.

3. Open valve HV04.

4. Weigh 0.5% of bleaching earth using the formula:

(density of palm oil at 90°C is 0.8561g/mL) , $\rho = \frac{Mass}{Volume}$

$$\frac{x\ g\ of\ Bleaching\ earth}{Mass\ of\ Neutralized\ palm\ oil\ (g)} \times 100 = 0.5\%$$

4. When temperature set point is achieved, add the weighed bleaching earth into R1 through hopper T3.

5. Close valve HV03, HV04, and HV14. Open valve HV13 (vacuum suction) and HV12 (sealant water).

6. Start vacuum pump L1 to create a vacuum of 0.4 bar abs (PT101) in reactor R1.

7. Set a timer for 30 minutes for the bleaching process starting at 90 °C.

G. Filtration

1. Reduce the set point of TIC102 to 40 °C. Maintain mixing at 200 rpm.

2. Stop vacuum pump L1.

3. Open valve HV14 to return R1 to atmospheric.

4. Open valve HV11 (cool down reactor) and close HV12 (vacuum pump ring sealant).

5. When the set point is reached, open HV06 and HV08 for multi-pass filtration. Ensure HV10 is closed.

6. Ensure sufficient pressure at PR01 (3 barg) and PR02 (2 barg), then start pump P1.

7. Allow filtration to proceed until clear oil is observed through the sight glass after the filter.

8. To collect the bleached palm oil (BPO), open HV09 and close HV08 to divert flow into feed tank T1.

9. Turn off heater W2 before emptying the reactor.

WARNING: Ensure heater W2 is switched off before draining the reactor R1 to prevent overheating.

10. End the experiment by performing shut down procedures as in H.

H. General Shut Down Procedures

1. Switch off the heaters (W1, W2).

2. Stop all pumps (L1, P1) and mixer (M1).

3. Ensure the reactor R1 is returned to atmospheric pressure by opening valve HV13 and HV14.

4. Fully drain the reactor R1 with HV07 and feed tank T1 with HV01.

5. Drain any leftover liquid in reagent tank T2 with HV03.

6. With a waste bucket prepared for filter S1 discharge, open the filter drain valve to vent any pressure and waste into the bucket.

7. Close HV08 and HV09.

8. Open HV10 to allow compressed air to blow through the filter to remove any filter cake layer. 9. Turn off the unit with the Main Switch at the control panel. 10. Close all valves after the unit is fully drained.
I. Housekeeping 1. Follow the instruction of your instructor.

Result

Yield Calculation

Component		**Volume (Liter)**	**FFA (%)**
A	Feed oil		
B	Processed Oil		

$$\text{Yield of NPO} = \frac{Volume\ of\ Refined\ Oil}{Volume\ of\ Feed\ Oil} \times 100$$

Discussion

1. Why is the determination of free fatty acid analysis is one of the oil quality parameter?
2. What factor that affects the on acid value in oil sample?
3. Compare the result before and after refining process? Explain your answer
4. What are the factors affecting the results?
5. State a few precautionary steps that should be followed in this experiment.

Questions

1. Give factors that affects the FFA contents in oils and fats.
2. How to reduce FFA content in oils? Explain your answer.

Discussion

1. What is the importance of color test in edible oils?
2. Discuss the results obtained and explain what is the factor affecting your results.
3. In this experiment, what is the error that may arise from color test?

Questions

1. What is the correlation between color test with refining process?
2. Some crude seed oils color tends to darken in storage. In your opinion, what is the possible attribute condition that may affect to the color of oils?
3. Besides Lovibond method, what are the methods employed in the determination of oil color?

Discussion

Provide a relevant discussion such as the methods principle, the important of the analysis, observation, data analysis and error that may arise from this experiment.

Conclusion

Provide a relevant conclusion based on this experiment.

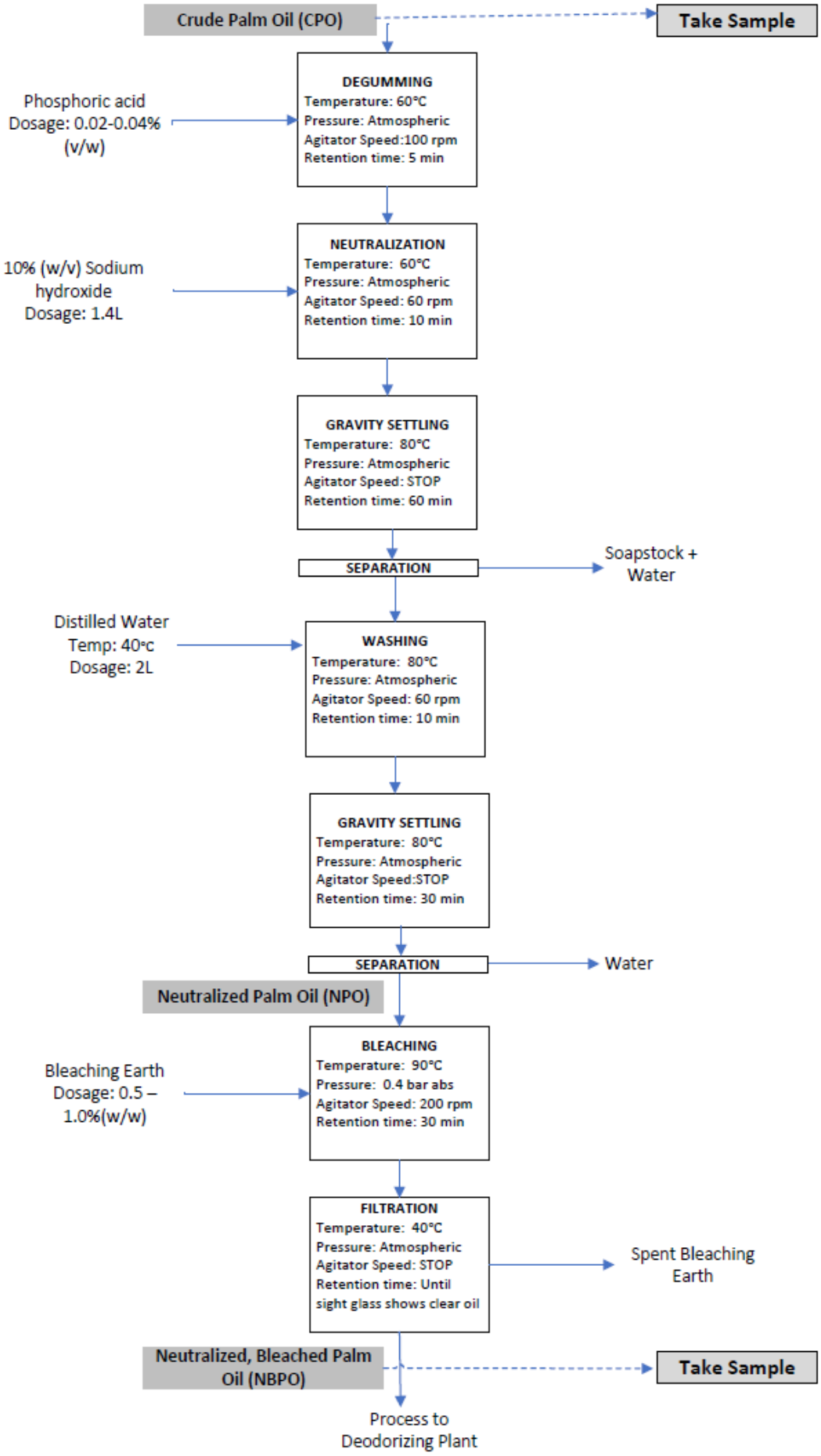
Questions

1. Describe the function of phosphoric acid dosing
2. Describe the principle of alkali/chemical refining
3. Why neutralization process must be done with low agitation speed?
4. Describe the reason for the water washing operation
5. Explain the usage of bleaching earth in bleaching

Reference

Provide the sources of reference for your discussion.

Process Flow Chart of the Oil Refining (Liquid) Pilot Plant



EXPERIMENT 5 : OIL REFINING - DEODORIZATION OF NEUTRALIZED AND BLEACHED PALM OIL

Objectives:

i. To carry out deodorization on neutralized and bleached palm oil in a batch process.
ii. To analyze and compare the quality of input and output oil from a batch refining.

Introduction:

Deodorization is the final step in vegetable oil processing in an oil refinery. The subsequent step is product storage, packing, and shipping. Therefore, the product leaving the deodorizer, at the final stage, should be ready for shipment. All finished product standards must be met, otherwise the product will be either reprocessed or returned by the industrial users for unsatisfactory quality, or worst there would be complaints on the brands from consumer. In any case, it is a costly affair for the company to respond to these customer complaints. Additionally, persistent customer complaints can lead to long-term loss of customers.

Deodorization, is a distillation process, using steam as carrier, in order to remove unwanted odour and taste from the degummed or neutralized oil for the purpose of producing high quality oil or tallow. Deodorization of fats and oils normally consists of steam distillation at elevated temperature under reduced pressure, although nitrogen has also been used. In reality, the process of deodorization performs numerous other functions as listed below:

- Reduces free fatty acid (FFA) to <0.05%, preferably <0.03%.
- Reduces the red and yellow colour in the refined and bleached (RB) oil and makes it lighter by heat bleaching in the deodorization process which decolorizes the carotenoids at high temperature under vacuum.
- Removes the odoriferous compounds, such as aldehydes, ketones, hydrocarbons, lactones, alcohol, etc. produced from decomposition of the oils.
- Reduces peroxide value (PV) to zero. At the same time, the anisidine value (pAV) increases.
- The oil loses a significant portion of the natural antioxidants (mostly tocopherols and some sterols).

- Any residual trace metals, picked up by the oil after bleaching, are reduced via citric acid treatment (chelation or scavenging process). This is an essential step and is not a substitute for the bleaching step.
- There is some increase in the amount of polymers, conjugated dienes, or other oil decomposition products.
- There can be a very small but detectable increase in the trans fatty acid content in the oil, depending on the deodorizer temperature.

The SOLTEQ-QVFTM Oil Deodorizer Unit (Model: BP516) is a lab-scale batch processing vessel capable of carrying out the deodorization process on small batches of edible oils, as part of the edible oil refining process.

Free fatty acids (FFA), ketone and acetaldehyde traces in edible oils render an unpleasant odour to the oils. Removal of these compounds is achieved by a steam stripping process under high temperature and high vacuum conditions, where an open steam is passed through the heated oil to carry away the evaporated volatile materials. Direct steam injection also provides a high degree of turbulence to the oil, ensuring good physical contact of the two phases. The oil is then cooled and passed through a filter to get sparkling oil, which is also bland and tasteless. Through this process, the peroxide value of the oil is brought down to a minimum.

Figure 3.1: Oil Deodorizer Unit (Model: BP516)

No.	Component	No.	Component
1	Reactor, R1	5	Control Panel
2	Pressure Filter, S1	6	Steam Ejector, J1
3	Feed Tank, T1	7	Vacuum Pump, L1
4	Transfer Pump, P1		

Material:

Neutralized, Bleached Palm Oil

Apparatus:

Measuring Cylinder 2L

Operating Procedures:

A. General Start-up Procedures

1. Ensure that heater W1 switch is in the off position on the control panel.
2. Power up the control panel with the Main Switch.
3. Ensure that all valves are initially closed.
4. Start the steam boiler. Open the steam trap bypass valve to drain leftover condensate. Close the bypass valve after a few minutes to allow steam pressure build up.

5. Check and if necessary, adjust the air pressure regulator PR01 to 3 kgf/cm2.

6. The unit is now ready for experiment.

B. Deodorization

1. Perform the general start-up procedures as described in Section 5.1.

2. Prepare 20 L of palm oil in feed tank T1.

3. Fully open HV11 to allow water into sealant ring of vacuum pump L1.

4. Open HV02 and HV10.

5. Start L1 to create a vacuum in reactor R1 and draw in the CPO from feed tank T1.

6. Close HV02 when T1 is empty. Allow vacuum pull down to maximum.

7. Set temperature of TIC101 for R1 to 180 °C. Turn on heater W1.

8. Once set temperature is achieved, begin steam introduction.

9. Set PR02 to 2 barg (PG301) and slightly open HV13 to charge steam into R1. Avoid addition of excess steam which will increase the reactor pressure.

10. Open HV14 and adjust PR03 to achieve steam pressure of 1 barg at PG303 into ejector J1.

11. Adjust HV13 and HV14 accordingly to maintain PT101 pressure below 0.1 barg.

12. Set a timer for 30 minutes.

13. When deodorization is complete, close both HV13 and HV14. Switch off heater W1.

14. Fully open HV12 to allow cooling water into the coils. Cool down contents in R1 to less than 50°C.

15. Return reactor R1 to atmospheric conditions by venting with HV09.

16. To collect product, open valve HV06.

17. Start pump P1 to pass the oil through filter S1 and into tank T1 as product. Adjust PR01 accordingly to increase pump speed.

WARNING: Ensure heater W1 switched is off before draining the reactor R1 to prevent overheating.

18. End the experiment by performing shut down procedures.

C. General Shut Down Procedures

1. Switch off the heater (W1).

2. Ensure steam supply valves are closed (HV13 and HV14).

3. Stop all pumps (L1 and P1). Allow sealant water into vacuum pump L1 to continue running to cool down.

4. Ensure the reactor R1 is returned to atmospheric pressure by opening valve HV09.

5. Fully drain the reactor R1 with HV06 and HV07; and feed tank T1 with HV01. 6. With a waste bucket prepared for filter S1 discharge, open the filter drain valve HV08 to vent any pressure and waste into the bucket. 7. Drain the sampling tube with HV03 and HV05. 8. Turn off the unit with the Main Switch at the control panel. 9. Close all valves after the unit is fully drained.
D. Housekeeping 1. Follow the instruction of your instructor.

Discussion

1. Why is the determination of free fatty acid analysis is one of the oil quality parameter?
2. What factor that affects the acid value in oil sample?
3. Compare the result before and after refining process? Explain your answer
4. What are the factors affecting the results?
5. State a few precautionary steps that should be followed in this experiment.

Questions

1. Give factors that affects the FFA contents in oils and fats.
2. How to reduce FFA content in oils? Explain your answer.

Discussion

1. What is the importance of color test in edible oils?
2. Discuss the results obtained and explain what is the factor affecting your results.
3. In this experiment, what is the error that may arise from color test?

Questions

1. What is the correlation between color test with refining process?
2. Some crude seed oils color tends to darken in storage. In your opinion, what is the possible attribute condition that may affect to the color of oils?
3. Besides Lovibond method, what are the methods employed in the determination of oil color?

Discussion

Provide a relevant discussion such as the methods principle, the important of the analysis, observation, data analysis and error that may arise from this experiment.

Conclusion

Provide a relevant conclusion based on this experiment.

Questions

1. Compare the quality parameter obtained before and after the deodorization process
2. Explain why deodorization must be operated in low pressure (vacuum)
3. Explain the purpose of steam stripping

Reference

Provide the sources of reference for your discussion.

EXPERIMENT 6: DRY FRACTIONATION OF RBD PALM OIL

Objectives:

1. To perform dry fractionation of refined, bleached and deodorized palm oil.
2. To determine the yield of olein and stearin products.
3. To analyze the quality parameter of modified fats.

Introduction:

Dry fractionation is the most commonly used fractionation process due to its simplicity and cost-effectiveness. Without the addition of other chemicals or additives, the triacylglycerol (TAG) crystals formed during the crystallisation process will be directly filtered to separate the solid and liquid fraction. However, some liquid will remain in the solid fraction due to entrapment. This method minimizes the product loss. Single-stage dry fractionation can produce olein with iodine value (IV) in the range of 56 to 62 (Gijs, et al., 2007). Double-stage or triple-stage dry fractionation can produce super olein with IV 65 and top olein with IV 70 (Kellens, et al., 2007).

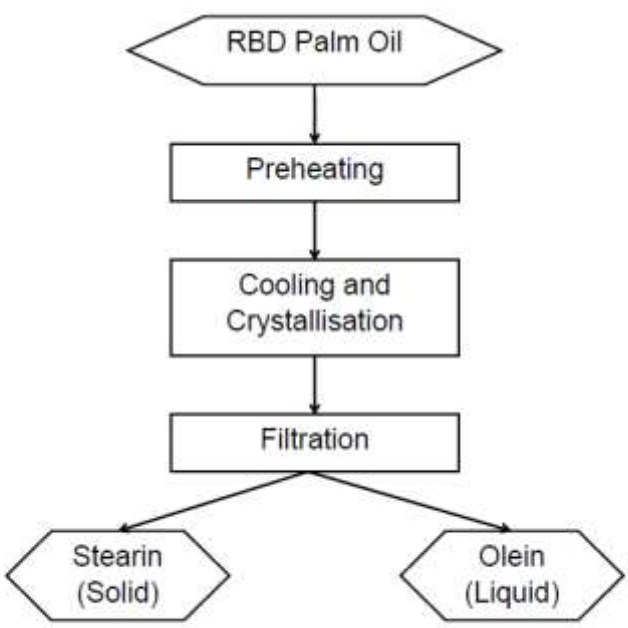


Figure 1: Process flow on the crystallisation of RBD palm oil

The Oil Fractionation Pilot Plant is instrumented and integrated with SCADA system (Supervisory control and data acquisition) to monitor the process variables in real time. The most critical parameter to be closely monitored is the temperature of crystallisation in the crystalliser. A precise and well-controlled temperature plays an important role in developing crystals of desired morphology and size as well as to ensure optimum stearin yield.

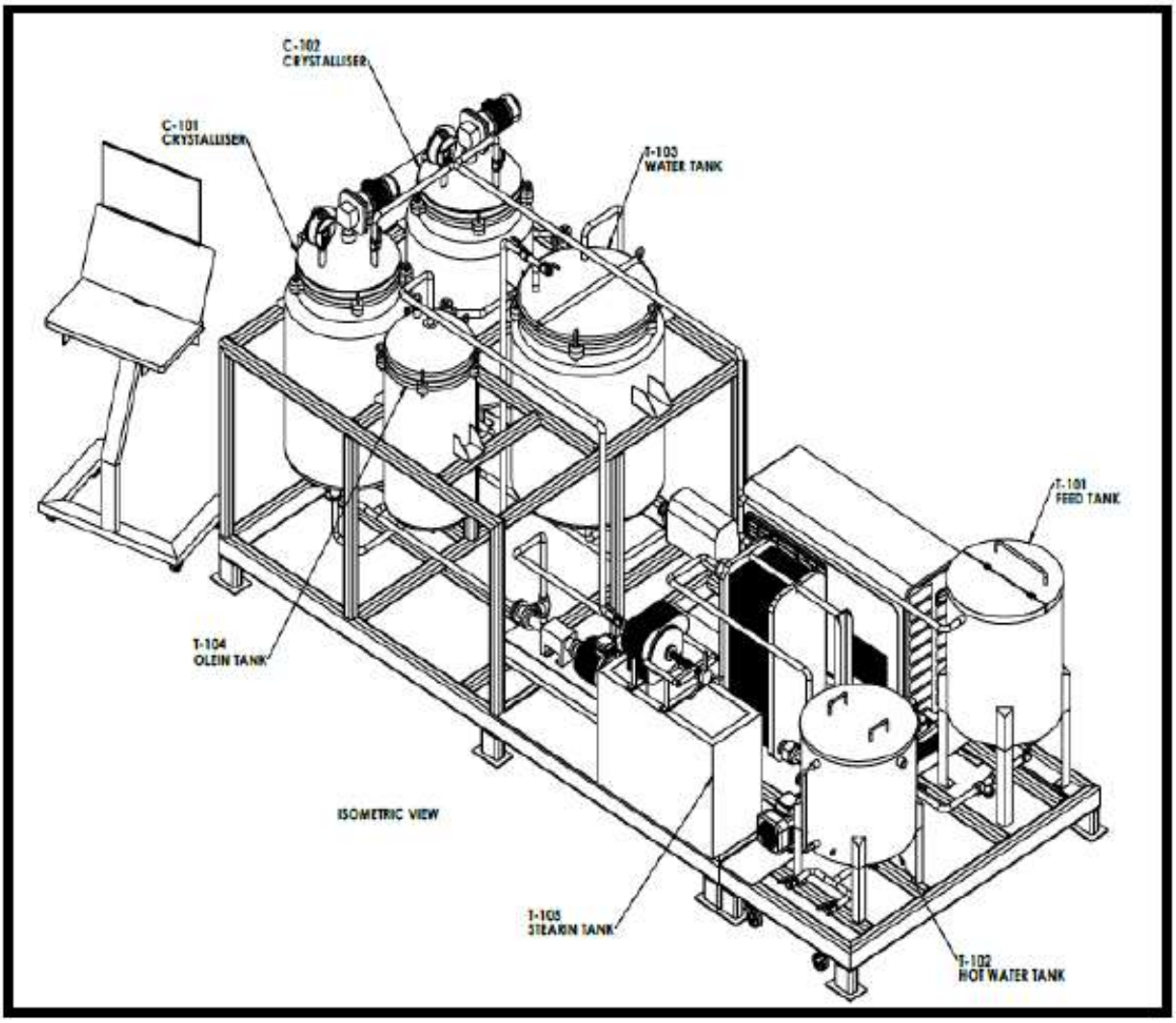


Figure 2: Oil Fractionation Pilot Plant

Steam and electric heaters are the sources of heating while the chiller is the source of cooling. A local control panel with switch buttons enables user to switch ON or OFF the electrical components for teaching and learning.

Material:

Refined, Bleached, Deodorized Palm Oil (RBDPO)

Apparatus

Weighing Balance

Operating Procedures:

A. General Start Up
1. Ensure that all power supply switch are turned off. 2. Perform a quick inspection on all electrical parts for damage or wear. 3. Check all electrical connections and other parts are secured correctly and fastenings are sufficiently tight. 4. Position the equipment safely on a solid, level surface. 5. Ensure the main plug is connected to power source. 6. Turn on the main power source. 7. All the indicators will light up. 8. Ensure all the hand valves, ball valves, globe valves and gate valves are closed before the experiment. 9. Fill in the water to T-102 and ensure water level is above the level switch low (LSL). 10. Fill in the water to T-103 and ensure water level is above the jacketed height. 11. The equipment is now ready to use.
B) Feed Tank Preparation
1. Fill T-101 with at least 50kg of RBD Palm Oil. 2. After Step 1, LSL-101 should not give Alarm. 3. Fill T-102 with tap water until approximately 25% full, or until heater (HTR-101) is fully immersed in the water. 4. After step 1, LSL-102 should not give Alarm. 5. Manually input the desired set point for HTR-101 in the range of 30 to 65C. 6. Click HTR-101 on SCADA, and switch ON HTR-101. 7. When LSL-102 does not give Alarm, HTR-101 is allowed to switch On.

8. When TE-102 gives temperature reading higher than set point of HTR-101, HTR-101 will **NOT** switch ON.

9. As HTR-101 is switched **ON**, TE-102 shows rising temperature through time. Wait until TE-102 achieves desired temperature.

10. When TE-102 achieves set point, HTR-101 automatically switched **OFF**.

C) Chill Water Tank Preparation

1. Fill T-103 with tap water until full (which is the same height as the jacket height).

2. T-103 must be fully filled with water to prevent icing on the refrigerant copper tubing and the refrigerant compressor.

3. Manually open MBV-113 and MBV-114.

D) Preheat Feed

1. Manually open MBV-104 and MBV-106.

2. Switch ON P-102.

3. Manually open MBV-101 and MBV-103.

4. Switch ON P-101.

5. TE-101 shows rising temperature through time. Wait until TE-101 achieves steady-state temperature.

6. Switch OFF P-102.

7. Manually close MBV-104 and MBV-106.

Or

For Steam Piping heating method*

1. Ensure electrical steam boiler is switched on with desired temperature pressure.

2. Manually open MBV-101 and MBV-103.

3. Switch ON P-101.

4. Manually open MBV-123 (Drain) and slowly turn MGV-101 to a quarter.

5. TE-101 shows rising temperature through time. Wait until TE-101 reach around 60-70C.

6. Manually Close MGV-101 and MBV-123 (Drain).

E) Oil Transfer from T-101 to C-101/C-102

1. User to select whether to operate with C-101 or C-102.

2. If user select C-101 in step 8, proceed with step 3-6; if user select C-102 in step 8, proceed with step 7-10.

3. Manually open MBV-108. If flowrate from T-101 to C-101 is slow, manually close MBV-103 to half open position.

4. Switch ON M-101.

5. When oil level in T-101 is low, the low-level alarm is automatically switched OFF P-101 to protect the pump from running dry.

6. Observe temperature reading on TE-104. TE-104 should show similar reading to TE-101 after the transfer.

7. Manually open MBV-110. If flowrate from T-101 to C-101 is slow, manually close MBV-103 to half open position.

8. Switch ON M-102.

9. When oil level in T-101 is low, the low-level alarm is automatically switched OFF P-101 to protect the pump from running dry.

10. Observe temperature reading on TE-105. TE-105 should show similar reading to TE-101 after the transfer.

F) Crystallisation (Crystallizer)

1. If user select to operate with C-101, proceed with step 2-11;if user select to operate with C-102, proceed with step 12-21.

2. Manually open MGV-103.

3. Switch ON P-103.

4. Observe temperature reading on TE-104 and TE-106. Both TE-104 and TE-106 should show rising temperature through time.

5. When TE-104 achieves desired temperature, switch OFF P-103 and hold the temperature for a desired duration to allow step-wise cooling.

6. Repeat step 2 to 5 until TE-104 achieves temperature close to 35°C.

7. User to manually input the desired set point for CH-101 in the range of 18 to 30°C.

8. User to click CH-101 on SCADA, and switch ON CH-101.

9. Wait until TE-103 achieves set point temperature.

10. Repeat step 2 to 5 until TE-104 achieves desired temperature of crystallizer. Switch OFF P-103 and hold the temperature for a desired duration before crystallization is complete.

11. Once crystallization in C-101 is complete, switch OFF M-101.

12. Manually open MGV-104.

13. Switch ON P-103.

14. Observe temperature reading on TE-105 and TE-106. Both TE-105 and TE-106 should show rising temperature through time.

15. When TE-105 achieves desired temperature, switch OFF P-103 and hold the temperature for a desired duration to allow step-wise cooling.

16. Repeat step 12 to 15 until TE-105 achieves temperature close to 35 C.

17. User to manually input the desired set point for CH-101 in range of 18 to 30 C.

18. User to click CH-101 on SCADA, and switch ON CH-101.

19. Wait until TE-103 achieves set point temperature.

20. Repeat step 12 to 15 until TE-105 achieves desired temperature of crystallizer. Switch OFF P-103 and hold the temperature for a desired duration before crystallization is complete.

21. Once crystallization in C-102 is complete, switch OFF M-102.

G) Slurry Transfer from C-101/C-102 to Product Tanks

1. If user select to operate with C-101, proceed with step 2-12, if user select to operate with C-102, proceed with step 13-23.

2. Ensure F-101 is fully sealed and tighten by rotating the handwheel clockwise.

3. Manually open MBV-109, MBV-118 and MBV-117.

4. Obtain the slurry from the outlet of MBV-115 and transfer it into the small cylinder until LSL-103 should not give alarm.

5. Switch ON P-104.

6. When P-104 is switched ON, liquid product enters T-104.

7. During slurry transfer, observe the pressure gauge on F-101. Once it achieve 0.4 bar, close P-104 immediately.

8. If LSL-103 gives alarm, P-104 will automatically switched off by default.

9. Drain both inlet and outlet pipeline from F-101 by opening the sampling valve to make sure all the sludge/olein are removed before loosen F-101.

10. Loosen F-101 by rotating the handwheel counter-clockwise.

11. Remove the cake sandwiched between the filter plates and filter media.

12. The cake will fall into T-105.

13. Ensure F-101 is fully sealed and tighten by rotating the handwheel clockwise.

14. Manually open MBV-111, MBV-118 and MBV-117.

15. Obtain the slurry from the outlet of MBV-115 and transfer it into the small cylinder until LSL-103 should not give alarm.

16. Switch ON P-104.

17. When P-104 is switched ON, liquid product enters T-104.

18. During slurry transfer, observe the pressure gauge on F-101. Once it achieve 0.4 bar, close P-104 immediately.

19. If LSL-103 gives alarm, P-104 is automatically switched off.

20. Drain both inlet and outlet pipeline from F-101 by opening the sampling valve to make sure all the sludge/olein is removed before loosen F-101.

21. Loosen F-101 by rotating the handwheel counter-clockwise.

22. Remove the cake sandwiched between the filter plates and filter media.

23. The cake will fall into T-105.

H) Stearin Transfer

1. Partially open MBV-122 until water overflows from the jacket of T-105.

2. Once water bath jacket is fully filled, manually close MBV-122.

3. User to manually input the desired set point for HTR-102 and HTR-103 in the range of 30 to 65C.

User to click HTR-102 or HTR-103 on SCADA, and switch ON HTR-102.

5. When TE-107 gives temperature reading higher than set point of HTR-102, HTR-102 will NOT switch ON.

6. As HTR-102 or/ and HTR-103 is switched ON, TE-107 shows rising temperature through time. Wait until TE-107 achieves desired temperature.

7. When TE-107 achieves set point, HTR-102 is automatically switched OFF.

8. Solid stearin product will melt into molten stearin that can be transferred out of T-105 by manually opening MBV-121.

I) General Start Down

1) Switch off the power supply.

2) Open MBV- 107 ,MBV-113 and MBV-112 to drain the water out from T-102 and T-103 respectively. 3) Open MBV-102, MGV-109, MGV-111 and MBV-115 to drain the remaining RBD palm oil out from T-101, C-101 and C-102 respectively. 4) Connect Flexible hose to MBV-119 and MBV-121 to remove Olein Product and Stearin Product.
J. Housekeeping 1. Follow the instruction of your instructor.

Result:

1. **Data collection from the cooling process (crystallization process) and sketch the cooling curve graph.**

Temperature (°C)	Time (s)	Observation

Note: time must be taken when temperature drop to 1 degree (°C).

2. **Sample yield table.**

Oil	Weight (kg)
RBD Palm Oil	
Stearin	
Olein	

$$\text{Yield of Olein} = \frac{Weight\ of\ Olein}{Weight\ of\ RBD\ Palm\ Oil} \times 100$$

$$\text{Yield of Stearin } = \frac{Weight\ of\ Stearin}{Weight\ of\ RBD\ Palm\ Oil} \times 100$$

Analysis Result

Refer your instructor on the analysis to be carried out and the laboratory manual.

Oil	Iodine Value
RBD Palm Oil	
Stearin	
Olein	

Discussion

1. Which sample shows the highest and lowest value? Explain the answer.
2. What is the importance of this analysis?
3. What is the reaction involved in this analysis?
4. Explain why do samples should be kept in the dark place?
5. State the precautionary steps that should be taken in this experiment.

Questions

1. What is the correlation between iodine value and the stability of fats and oils?
2. Explain how iodine value is used to monitor the progress of hydrogenation.
3. Besides the iodine value, name other methods used in industry to monitor hydrogenation.

Discussion

Provide a relevant discussion such as the methods principle, the important of the analysis, observation, data analysis and error that may arise from this experiment. Sketch the cooling cur graph base on data collection.

Conclusion

Provide a relevant conclusion based on this experiment.

Questions

1. Explain why preheating must be done before the crystallization process.
2. Explain the changes of iodine value between input oil (RBDPO) and output oil (Stearin and Olein)
3. Explain how the olein is being separated from the stearin fraction.

Reference

Provide the sources of reference for your discussion.

EXPERIMENT 7: HYDROGENATION OF UNSATURATED PALM OIL

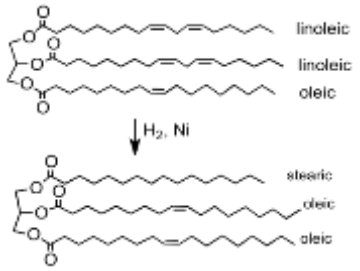
Objectives:

To carry out hydrogenation on unsaturated palm oil in a batch process.

Introduction:

Hydrogenation of vegetable oil has been practiced for over a century. The process was originally introduced to convert some of the unsaturated fatty acids in vegetable oils, as well as marine or animal fats to make them more stable to oxidation. In this process, the unsaturated double bonds in the fatty acids of the oil molecules react with hydrogen atoms in the presence of a catalyst. Nickel catalyst is used in commercial hydrogenation of edible oils. Other catalysts, such as platinum, palladium, copper, etc., have also been applied in hydrogenation applications. However, they are are not used in commercial hydrogenation of edible oils.

Hydrogenation has been used for a long time to improve oxidative stability of vegetable oils for an improved shelf life. It has also been carried out to modify the solids content and melting characteristics of the oil to formulate shortening and margarine products with the desired physical properties. Diagram below illustrates addition of hydrogen to a hydrocarbon chain, breaking the double bonds and essentially saturating the chain.



Hydrogenation is a heterogeneous reaction process and complex in nature where the reaction occurs between the hydrogen (gaseous phase) and the unsaturated fatty acids (liquid phase), converting some or all of the unsaturated fatty acids into saturated fatty acid (stearic acid). This involves the following physical and chemical reactions:

- Mixing and dispersion of the catalyst by a mechanical mixer.
- Diffusion of hydrogen gas through the mass of oil to the catalyst surface.

- Adsorption of the reactants (hydrogen and unsaturated fatty acids) on the catalyst surface.
- Partial or complete saturation of the unsaturated fatty acids on the catalyst surface.
- Desorption of the products reaction and the unreacted fatty acids and oil molecules from the catalyst surface.
- Release of heat due to the exothermic nature of the hydrogenation reaction.

Hydrogen gas and the unsaturated fatty acids diffuse through the bulk of the oil and reach the catalyst surface. Migration of these reactants through the bulk of the oil is facilitated by mechanical agitation. The two reactants, hydrogen and the unsaturated fatty acids, diffuse through a stagnant microfilm of oil on the surface of the catalyst to reach the active sites on the catalyst. The reaction between the unsaturated fatty acids and hydrogen gas occurs on the catalyst surface. This is sometimes referred to as the chemisorption process, implying a chemical reaction aided by the adsorption process. The products of the reaction, which are typically saturated fatty acids, unsaturated fatty acids (cis and trans isomers), and triglyceride molecules, leave the catalyst surface via the desorption process. More fresh reactants are adsorbed on the surface of the catalyst and the hydrogenation reaction continues. Heat is also generated in the reaction.

The SOLTEQ-QVFTM Oil Hydrogenation Unit (Model: BP517) is a lab-scale batch processing vessel capable of carrying out an oil modification process – hydrogenation, on small batches of edible oils, to increase the degree of saturation.

Most oils are liquid at room temperature, which can be transformed into a semi-liquid or solid form for different applications and improve their functionality. By varying the process conditions, unsaturated oils can be hardened by reacting with hydrogen gas, in the presence of a nickel catalyst to speed up the rate of reaction, forming saturated fats.

Figure 3.1: Oil Hydrogenation Unit (Model: BP517)

No.	Component	No.	Component
1	Catalyst Tank, T2	5	Stirrer Motor, M1
2	Reactor, R1	6	Control Panel
3	Pressure Filter, S1	7	Vacuum Pump, L1
4	Feed Tank, T1	8	Transfer Pump, P1

Material:

Unsaturated Palm Oil (RBD Palm Oil), Nickel Catalyst

Apparatus:

Measuring Cylinder 2L, Analytical Balance, Beaker 100Ml

Operating Procedures:

A. General Start-up Procedures

1. Power up the control panel with the Main Switch.
2. Ensure that all valves are initially closed.
3. Start the steam boiler. Open the steam trap bypass valve to drain leftover condensate. Close the bypass valve after a few minutes to allow steam pressure build up.
4. Check and if necessary, adjust the air pressure regulator PR01 and PR03 to 3 kgf/cm2.
5. The unit is now ready for experiment.

B. Hydrogenation

1. Perform the general start-up procedures as described in Section A.
2. Prepare 18 L of unsaturated palm oil in feed tank T1.
3. Prepare 2 L of unsaturated palm oil in catalyst tank T2. Mix in 20 g of catalyst.
4. Open HV15 and adjust PR03 and PR05 to mix the catalysts with oil in T2.
5. Fully open HV16 to allow water into sealant ring of vacuum pump L1.
6. Open HV02 and HV12.
7. Start L1 to create a vacuum in reactor R1 and draw in roughly 10 L of oil from feed tank T1.
8. Close HV02 and open HV03. Allow the catalyst-oil suspension to be drawn into R1.
9. When T2 is empty, close HV03 and open HV02 again to draw in the remaining oil from T1.
10. Close HV02 when T1 is empty and the allow vacuum pull down to maximum.
11. Open HV14 and adjust PR04 to achieve mixing speed of around 150 rpm.
12. Adjust HV18 for nitrogen gas purging.
13. Stop vacuum pump L1.
14. Open HV19 slightly to pressurize the vessel back to atmospheric (1 bar atm).
15. Close HV19, and start vacuum pump L1 again to allow vacuum suction for 5 minutes.
16. Repeat steps 13 to 15 for an additional 2 times.
17. After completing the third nitrogen purge, close HV12 to maintain the vacuum in R1.
18. Stop L1 and close HV16.
19. Change direction of valve HV18 to sparge in hydrogen gas.
20. Open HV19 slowly to fill R1 and pressurize to around 3 bar abs. (PT101).
21. Begin to heat up the reaction by introducing steam into the heating coils. Open the steam trap bypass valve to drain leftover condensate.

22. Open the steam inlet valve and adjust PR06 to achieve around 3 barg of steam in the coils.

23. Once steady state is achieved around 130 to 140°C, start a timer for 2 hours.

24. When hydrogenation is complete, close the steam supply valve.

25. Fully open the cooling water valve HV17. Cool down contents in R1 to less than 50°C.

26. Return reactor R1 to atmospheric conditions by venting with HV09.

27. Adjust HV18 for nitrogen gas purging.

28. Open HV19 to pass nitrogen gas into R1 continuously for 5 minutes to displace and remove hydrogen gas in the reactor. Ensure pressure in R1 do not exceed 3 bar abs. at PT101.

29. Close HV19 and allow the reactor to vent back to atmospheric pressure.

30. Open HV05 and HV10.

31. Start pump P1 to pass the product through filter S1 to separate the catalysts, and collect the saturated product in T1. Adjust PR02 if necessary to increase flow rate.

32. Stop P1 when reactor R1 is emptied.

33. End the experiment by performing shut down procedures as in section C.

C. General Shut Down Procedures

1. Ensure steam supply valve is closed.

2. Close HV14 and HV15 to stop the stirrers.

3. If reactor R1 is still pressurized with hydrogen gas, slowly open HV11 to vent. Ensure HV19 is closed.

4. When reactor is back to atmospheric (1 bar abs), purge R1 by introducing nitrogen gas. Switch HV18 to nitrogen and open HV19. Keep vent HV11 open.

5. After purging for 5 minutes, close HV19.

6. Fully drain the reactor R1 with HV05 and HV06; and feed tank T1 with HV01.

7. With a waste bucket prepared for filter S1 discharge, open the filter drain valve HV08 to vent any pressure and waste into the bucket.

8. Open HV07 to allow compressed air to blow through the filter to remove the catalysts.

9. Turn off the unit with the Main Switch at the control panel.

10. Close all valves after the unit is fully drained.

D. Housekeeping

1. Follow the instruction of your instructor.

Discussion

1. Which sample shows the highest and lowest value? Explain the answer.
2. What is the importance of this analysis?
3. What is reaction involved in this analysis?
4. Explain why do samples should be kept in the dark place?
5. State the precaution steps should be taken in this experiment.

Questions

1. What is the correlation between iodine value and the stability of fats and oils?
2. Explain how iodine value is used to monitor the progress of hydrogenation.
3. Besides the iodine value, name other methods that is used in industry to monitor hydrogenation.

Discussion

Provide a relevant discussion such as the methods principle, the important of the analysis, observation, data analysis and error that may arise from this experiment. Sketch the cooling cur graph base on data collection.

Conclusion

Provide a relevant conclusion based on this experiment.

Questions

1. Compare the degree of unsaturation of feed oil and hydrogenated oil
2. Explain the reason why hydrogenation process must be done in vacuum condition
3. Explain the function of nickel catalyst

Reference

Provide the sources of reference for your discussion.

EXPERIMENT 8 : DETERMINATION OF OIL BLENDING FOR COOKING OIL

Objectives:

i. To carry out oil blending experiment.
ii. To indicate the cloud point value of oil blending sample.
iii. To analyze and compare the quality of oil blending sample with the standard.

Introduction;

Oil blending is mixing of two or more ingredients to achieve the preset objectives. In this experiment the ingredients are vegetable oils. In blending, composite ingredients share advantages and disadvantages according to the blend ratio. Blending is commonly used to produce general purpose cooking and frying oils aimed at improved and extended physical, chemical and functional properties. It is important that all food and blending laws and guidelines are met in addition to the consumers expectations. Cooking oil is the product of blending of pemissible edible oils of vegetable origin which shall be refined, bleached and deodorized so as to conform to the given standard of quality for cooking oil as reproduced. Possible blending objectives are categorized as commercial,technical, functional and nutritional. Common oils used for blending are palm oleins, canola oil, rapeseed oil, soybean oil, sunflower oil and cottonseed oil.

Material:	**Apparatus:**
Palm Oleins Oil	50ml beaker
Coconut Oil	Glass rod
Olive oil	Spatula
	Dropper

Procedure:

1. Weigh of all oil sample according to Table 1.
2. Pour the coconut oil or olilve oil into the palm oleins oil.
3. Heat and stir the blending oil for 15 minutes at 70°C.
4. After 15 minutes, cool the blending oil and proceed with Cloud Point Analysis.

Table 1 : Blending oil ratio

No.	Palm oil volume (ml)	Coconut / Olive Oil volume (ml)
1	40	10
2	30	20
3	20	30
4	10	40

Discussion

1. Which sample shows the highest and the lowest value? Explain the answer.
2. What is the importance of this analysis?
3. What is the reaction involved in this analysis?
4. Explain why should the samples be kept in the dark place?
5. State the precautionary steps that should be taken in this experiment.

Questions

1. Describe the purpose of oil blending.
2. What factor that affects the on cloud point of the oil?
3. How can the problem in (2) be solved?
4. List a few approaches or methods that can be used to prevent the cloudy in oil.
5. What is the correlation between iodine value and stability of fats and oils?
6. Explain how iodine value can be used to monitor the hydrogenation process.
7. Besides the iodine value, give another method to monitor the above process.

Conclusion

Provide a relevant conclusion based on this experiment.

Reference

Provide the sources of reference for your discussion.

Analysis Method

Determination of Moisture Content – Oven Method [*: AOCS Ba 21-38*]

Objectives

To determine the moisture in the specified products, and any material that is volatile under the conditions of the test.

Introduction

Moisture content is the loss in mass percentage when the sample is drying at a certain temperature in the oven at atmospheric until practically constant mass is reached.

In oven drying method, the sample is heated under specific conditions (temperature, pressure, time) and the lost of weight is used to calculate the moisture content of the sample. The moisture content value obtained is highly dependent on the type of oven used, condition in the oven, time and temperature of drying.

Apparatus/Instruments

Aluminum moisture dish/aluminum pan/petri dish

Drying oven

Analytical balance

Desiccator

Chemical/Reagents/Samples

Fat samples

Procedures

1. Weigh about 5g (2g for soybean oil cake or meal or linseed meal) of the test sample into the tared moisture dish.
2. Place the dish in the oven and dry at ± 130ºC for two hours.
3. Remove from the oven, cover immediately, cool in a desiccator to room temperature and weigh.

Calculation of the Results

$$\text{Moisture content, \%} = \frac{\text{loss in mass,g}}{\text{mass,g of test portion}} \text{ X } 100$$

Sample	Mass of dish,g	Mass of test portion,g	Mass of sample after oven dried	Loss in mass, g	Moisture content, %
A	1.				
	2.				
B	1.				
	2.				
C	1.				
	2.				

Determination Of Free Fatty Acid In Oil Sample [*AOCS Ca 5a-50]*

Objectives

To perform free fatty analysis exists in sample.

Introduction

Free fatty acids (FFA) in plant oils and fats (e.g., edible oils and fats) are a quality feature for these fats. Fats with high levels of FFA are more susceptible to oxidative aging, they become rancid more quickly. The FFA should be removed during a refining process. The method is suitable for edible fats and oils such as butter, olive, palm or sunflower oil. The acid number is the quantity of base, expressed in milligrams of potassium hydroxide, which is required to neutralize all acidic constituents present in 1 g of sample. The calculation of FFA percentage depends on the type of titrated sample and the fatty acid to which the result is to be calculated.

Apparatus	**Chemical/Reagents**
Oil sample bottles	Vegetable oils/Marine oils/animal fats
Erlenmeyer flask	Ethyl alcohol – 95%
Burette	Phenolphthalein indicator solution – 1% in 95% alcohol
Hot plate	Sodium hydroxide solution – accurately standardized. See **Table 1** for
Analytical balance	the appropriate normality of the sodium hydroxide solution, depending on the expected free fatty acid concentration range in the sample.

Procedures

1. Test samples must be mixed well to be entirely liquid before weighing; however, do not heat the sample more than 10 ºC over the melting point.
2. Use **Table 1** to determine test portion weight for various ranges of fatty acids. Weigh the designated sample size into an oil sample bottle or Erlenmeyer flask.
3. Add specified amount of hot neutralized alcohol and 2 mL of phenolphthalein indicator.
4. Titrate with standard sodium hydroxide, shake vigorously until the appearance of the first

permanent pink color of the same intensity as that of neutralized alcohol before the titration addition of the sample. The color must persist for 30 seconds.

5. Repeat the step 1- 4 for 2 times for replication.

Table 1: Free fatty acid range, alcohol volume and strength of alkali

FFA range (%)	Test portion (g)	Alcohol (mL)	Strength of alkali
0.00 to 0.2	56.4 ± 0.2	50	0.1 M
0.2 to 1.0	28.2 ± 0.2	50	0.1 M
1.0 to 30.0	7.05 ± 0.05	75	0.25 M
30.0 to 50.0	7.05 ± 0.05	100	0.25 or 1.0 M
50.0 to 100.0	3.525 ± 0.0001	100	1.0 M

Calculations

1. The percentage of free fatty acids in most types of fats is calculated as oleic acid, although in coconut and palm kernel oils it is frequently expressed as lauric acid and palm oil in terms of palmitic acid.

a) Free fatty acids as oleic, % = $$\frac{\text{mL of alkali x M x 28.2}}{\text{mass,g of test portion}}$$

b) Free fatty acids as lauric, % = $$\frac{\text{mL of alkali x M x 20.0}}{\text{mass,g of test portion}}$$

c) Free fatty acids as palmitic, % = $$\frac{\text{mL of alkali x M x 25.6}}{\text{mass,g of test portion}}$$

2. The free fatty acids are frequently expressed in terms of acid value instead of percentage of free fatty acids. The acid value is defined as the number of milligrams of KOH necessary to

neutralize 1 g of sample. To convert the percentage of free fatty acids to acid value, multiply the percentage of free fatty acids by its conversion factor.

Conversion factor:

Palmitic = 2.19, Lauric = 2.81, Oleic = 1.99

Determination Of Iodine Value of Fats And Oils –

Cyclohexane Method [*AOCS Cd 1b-87]*

Objectives

To perform iodine value analysis using Cyclohexane method in fats and oils.

Introduction

The iodine value (IV) is a measure of the unsaturation degree of fats and oils that is related to the double or triple bonds of fats. It is expressed as the number of centigrams of iodine absorbed per gram sample (% iodine absorbed).

A solution of iodine monochloride (ICl, Wijs reagent) in a mixture of acetic acid and cyclohexane is added to a quantity of fat or oil. The amount of the iodine (iodine monochloride) left at the end of the reaction is then measured after reduction by adding potassium iodide solution and water. The liberated iodine is titrated with standard sodium thiosulphate (Na2S2O3) solution as an indicator.

The higher the amount of unsaturation, the more the iodine absorbed; therefore, the higher the iodine value, the greater the degree of unsaturation. This method is applicable to all normal fats and oils that are not containing conjugated double bonds. Iodine value (IV) is normally to characterize fats and oils, to monitor hydrogenation process as an indication of lipid oxidation since IV reduces as oxidation progresses.

Apparatus

Conical flask

Volumetric flak – 1000 mL

Pipet – 25 mL

Measuring cylinder – 5 mL, 50 mL

Repeater pipet – 20 mL (for cyclohexane)

Analytical balance

Filter paper

Beaker – 50 mL

Oven/Hotplate

Timer

Thermometer

Chemical/Reagents

Wijs solution

Cyclohexane

Soluble starch solution

Potassium dichromate ($K_2Cr_2O_7$)

Sodium thiosulphate ($Na_2S_2O_3.5H_2O$

Procedures

1. Preparing of solutions:
   a. Starch indicator solution (freshly prepared)
      - 1g of soluble starch and add small amount of cold water. Add, while stirring to 200 mL of boiling water.
   b. Sodium thiosulphate ($Na_2S_2O_3.5H_2O$), 0.1N
      - dissolve 24.9g of sodium thiosulphate in deionized water and dilute to 1 L.
2. Melt the sample if it is not in liquid state. (The melting temperature should not exceed the melting point of the sample more than 10 ºC).
3. Filter through two pieces of filter paper to remove any solid impurities and the last traces of moisture. the filtration may be formed in an oven at 80 – 85 ºC, but should be completed within 5 ± 30 sec. The sample must be dry.
4. After filtration, allow the filtered sample achieves a temperature of 68 – 71 ± 1 ºC before weighing the sample.

Sample weights.

Iodine value	Mass of sample 100% excess	Mass of sample 150% excess	Weighing accuracy
	g	g	g
<3	10	10	± 0.001
3	10.576	8.4613	0.005
5	6.346	5.0770	0.0005
10	3.1730	2.5384	0.0002
20	1.5865	1.2720	0.0002
40	.7935	.6346	0.0002
60	.5288	.4231	0.0002
80	.3966	.3173	0.0001
100	.3173	.2538	0.0001
120	.2644	.2115	0.0001
140	.2266	.1813	0.0001
160	.1983	.1587	0.0001
180	.1762	.1410	0.0001
200	.1586	.1269	0.0001

5. Once the sample achieved a temperature of 68 – 71 ± 1 ºC, immediately weigh the sample into a 500 mL conical flask.
6. Add 20mL of cyclohexane on top of the sample and swirl to ensure that the sample is completely dissolved.
7. Dispense 25 mL of Wijs solution using pipet into the flask containing the sample. Close the conical flask and swirl to ensure an intimate mixture. Immediately set the timer for for 1.0 or 2.0 hours depending on the iodine value of the sample: IV <150, 1.0hr; IV ≥ 150, 2.0hr.
8. Store the flask in the dark at temperature of 25 ± 5 ºC.
9. Prepare and conduct at least one black determination with each group of samples simultaneously and similarly to the samples respectively.
10. Titrate with 0.1 N $Na_2S_2O_3$ solution, by adding it gradually and with constant and vigorous

shaking. Continue the titration until the yellow color is almost disappeared. Add 1 – 2 mL of start indicator and continue the titration until the blue color disappears.

Calculations

The iodine value $= \frac{(B-S) \text{ x } N \text{ x } 12.69}{\text{mass, g of sample}}$

Where – B = volume of titrant, mL of blank

S = volume of titrant, mL of sample

N = normality of $Na_2S_2O_3$ solution

Determination of Iodine Value Of Fats And Oils – Wijs Method [*AOCS Cd 1-25]*

Objectives

To perform iodine value analysis using Wijs method in fats and oils.

Introduction

The iodine value (IV) is a measure of the unsaturation degree of fats and oils that is related to the double or triple bonds of fats. It is expressed as the number of centigrams of iodine absorbed per gram sample (% iodine absorbed).

Fats and oils reacted with an amount of measured iodine. In this halogenation reaction, iodine react and absorbs by the double bond (C=C) and the remaining iodine is calculated to identify the amount of absorbed iodine. The degree of unsaturation in fats and oils depends on the amount absorbed iodine.

In Wijs method, iodine monochloride (ICl) acts as halogenation agent. Iodine monochloride is added in a mixture of acetic acid and carbon tetrachloride. The ICl left or remained after a specific time is reduced by adding potassium iodide (KI) solution and water followed by titration of the liberated iodine with the standard sodium thiosulphate ($Na_2S_2O_3$) using starch solution as an indicator.

Apparatus	**Chemical/Reagents**
Conical flask	Wijs solution
Volumetric flask – 1000 mL	Carbon tetrachloride(CCl_4) – If unavailable, can
Pipet – 25 mL	bereeplace with mixture cyclohexane-acetic
Measuring cylinder – 5 mL, 50 mL	acid 1:1, v/v
Repeater pipet – 20 mL (for cyclohexane)	Soluble starch solution
Analytical balance	Potassium dichromate ($K_2Cr_2O_7$)
Filter paper	Sodium thiosulphate ($Na_2S_2O_3.5H_2O$)
Beaker – 50 mL	Soluble starch solution
Oven/Hotplate	
Timer	
Thermometer	

Procedures

1. Preparing of Solutions
    a. Starch indicator solution (freshly prepared)
        - 1g of soluble starch and add small amount of cold water. Add, while stirring to 100 mL of boiling water.
    b. Sodium thiosulphate ($Na_2S_2O_3.5H_2O$), 0.1N
        - dissolve 24.9g of sodium thiosulphate in deionized water and dilute to 1 L.

Sample weights.

Iodine value	Mass of sample 100% excess	Mass of sample 150% excess	Weighing accuracy
	g	g	g
<3	10	10	± 0.001
3	10.576	8.4613	0.005
5	6.346	5.0770	0.0005
10	3.1730	2.5384	0.0002
20	1.5865	1.2720	0.0002
40	.7935	.6346	0.0002
60	.5288	.4231	0.0002
80	.3966	.3173	0.0001
100	.3173	.2538	0.0001
120	.2644	.2115	0.0001
140	.2266	.1813	0.0001
160	.1983	.1587	0.0001
180	.1762	.1410	0.0001
200	.1586	.1269	0.0001

2. Melt the sample if it is not in liquid state. (The melting temperature should not exceed the melting point of the sample more than 10 ºC).
3. Filter through two pieces of filter paper to remove any solid impurities and the last traces of moisture. The filtration may be formed in an oven at 80 – 85 ºC, but should be completed within 5 ± 30 sec. The sample must be dry.
4. After filtration, allow the filtered sample achieves a temperature of 68 – 71 ± 1 ºC before weighing the sample.
5. Once the sample achieved a temperature of 68 – 71 ± 1 ºC, immediately weigh the sample into 500 mL conical flask.
6. Add 15 mL of carbon tetrachloride on top of the sample and swirl to ensure the sample is

completely dissolved.

7. Dispense 25 mL of Wijs solution using pipet into the flask containing the sample. Close the conical flask and swirl to ensure an intimate mixture. Immediately set the timer for 30 minutes.
8. Immediately store the flask in the dark for the required reaction time.
9. Remove the flasks from storage and add 20 mL of KI solution, followed by 100 mL of deionized water.
10. Titrate with 0.1N $Na_2S_2O_3$ solution, by adding it gradually and with constant and vigorous shaking. Continue the titration until the yellow color is disappeared. Add 1 – 2 mL of start indicator and continue the titration until the blue color disappears.
11. Prepare and conduct at least one black determination with each group of samples simultaneously and similarly aspect to the samples.

Calculations

$$\text{The iodine value} = \frac{(B-S) \text{ x } N \text{ x } 12.69}{\text{mass, g of sample}}$$

Where –

B = volume of titrant, mL of blank

S = volume of titrant, mL of sample

N = normality of $Na_2S_2O_3$ solution

Discussion

1. Which sample shows the highest and the lowest value? Explain the answer.
2. What is the importance of this analysis?
3. What is the reaction involved in this analysis?
4. Explain why should do samples be kept in the dark place?
5. State the precautionary steps that should be taken in this experiment.

Questions

1. What is the correlation between iodine value and stability of fats and oils?
2. Explain how iodine value can be used to monitor the hydrogenation process.
3. Besides the iodine value, give another method to monitor the above process.

Determination of Color In Oil Samples Using Lovibond Tintometer Color Scale

[AOCS Cc 13e-92]

Objectives

To determine the color intensity of oil by passing a beam of light from a light source through the oil and measuring the transmitted light with a silicon photo detector.

Introduction

Color is an important indication of the product composition, purity, and degree of deterioration. It is a quick check on degradation and the suitability and stability of the product for a particular use. In the case of vegetable oils, it is necessary to monitor each stage of the refining process to determine whether the required color has been obtained, as each type of oil will have its own "sell by color" specification.

Frequent color measurements are often the key to considerable savings in time and bleaching earths that are used for refining vegetable oils. Hence, color measurement is used for quality monitoring, production control, and determination of final product conformance to predetermined color tolerance and of compliance with customer specifications.

Visual methods are not always adequate for investigative purposes. For an objective and unbiased assessment, automated colorimeters are called for. These instruments are usually designed to measure the transmitted color of optically clear liquids. For obvious reasons, the precision of color measurements with automatic colorimeters is better than that for manual operated instruments.

Apparatus/Instruments

Lovibond Tintometer Model F

Lighting cabinet

Color racks

Spillage tray

Glass cells

Chemical/Reagents

Oil samples

Procedures

1. Preparation of Test Sample:
    - Fats or oils is completely liquid, clear and bright when determination is made. Heating shall be avoided if it is likely to cause a color change. If the test sample is not liquid at room temperature, heat it to a temperature about 10 ºC above melting point.
2. It is essential that the determination be carried out in subdued ambient light, i.e., not facing window or in direct sunlight.
3. Pour the prepared test sample into a cell sufficient optical path length to give color readings within the ranges given. Ensure that the cell is thoroughly clean and dry and, if necessary, prewarmed so that no solid matter separates from the test sample during the determination.
4. Place the cell containing the test portion in the lighting cabinet closed to the viewing tube. Immediately determine the color of the test portion, initially by using the color racks in the ration 10 yellow to 1 red and using the minimum number of blue or neutral units to obtain the match, but not more than 9.0 blue or 3.0 neutral.
5. Because the onset of eye fatigue is rapid, the operator shall rest the eyes after each 30 seconds period of matching. The test must be carried out by two trained operators.
6. If the requirements noted in Repeatability Section and Table 1 are not satisfied, a third trained operator must carry out the test. The mean of the two closest readings (of three) should be taken.
7. Record the size of the cell used and the red, yellow, blue or neutral readings forming the color match.

REPEATABILITY

The difference between two test results on the same material, in the same laboratory, under the same conditions, should not exceed the repeatability value, *r*.

Table 1 The results of an International test held by FOSFA International (values in Lovibond units).

Color scale 133.4mm cell	Palm oil (RBD)		Crude palm kernel oil	
	Red	Yellow	Red	Yellow
n	9	9	9	9
Mean (in Lovibond units)	2.30	21.60	5.00	47.7
Repeatability				
Sr	0.07	1.22	0.25	2.35
r	0.20	3.42	0.71	6.58

Expression of Results

1. Take the mean of the results obtained by two trained operators. If the requirements of repeatability (see Repeatability Section and Table 1) are not met, the mean of the closest readings (of three) should be taken.
2. Express the results in terms of the following.
   a. the number of red, yellow and blue or neutral readings needed to obtain the match.
   b. the length of the cell used
3. Color measurements taken in one cell length shall not be used to calculate the color values for another cell length.

Cloud Point Analysis [AOCS Official Method Cc 6-25]

Definition:

The cloud point is temperature at which, under conditions of this test, a cloud is induced in the sample caused by the first stage of crystallization.

Apparatus:

Sample bottle (120ml) or conical flask, thermometer and water bath.

**water bath: made up of water, chipped ice and water; or chipped ice, salt and water, depending on temperature required. The temperature of the cloud point bath shall not be less than 2^{o}C, or more than 5^{o}C below the cloud point. Either a beaker or insulated container is convenient for the test.

Sample: Palm oil and sunflower oil

Procedure

1. The sample must be completely dry before conducting the test. If the sample contains traces of moisture, it should be filtered through suitable filter paper. Heat 60-75 g of sample to 130°C just before conducting the test. Pour 45 mL of the heated fat into the oil sample bottle.
2. Begin to cool the bottle and contents in the water bath, stirring just enough to keep the temperature uniform. When the sample reaches a temperaturea of about 10°C above the cloud point, begin stirring steadily and rapidly in a circular motion so as to prevent supercooling and solidification of fat crystals on the sides or bottom of the bottle.
3. From this point on, do not remove the thermometer from the sample; doing so it may form air bubbles which will interfere with the test. The test bottle is maintained in such a position that the upper level of the sample in the bottle is level with the water in the bath.
4. Remove the bottle from the bath and inspect regularly. The cloud point is the temperature at which that portion of the thermometer immersed in the oil is no longer visible when viewed horizontally through the bottle and sample.
5. Record the time in Table 1.
6. Repeat the step 1-4 for replication.

Table 1 : Cloud Point Table

No.	Palm oil volume (ml)	Coconut / Olive Oil volume (ml)	Cloud point (°C) (Replication 1)	Cloud point (°C) (Replication 2)	Cloud point (°C) (Replication 3)	Average Cloud point (°C)
1	40	10				
2	30	20				
3	20	30				
4	10	40				

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-Robert Kiyosaki

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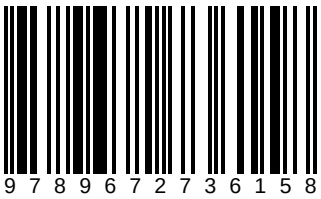

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