EXPERIMENTAL EXAMINATION
December 8, 2010

Abuja, Nigeria

Important Remarks

The three tasks are independent. Students in each team can decide to work cooperatively or separately.

1. While you are in the Venue of the Examination, you should wear safety spectacles at all times.
2. Eating of any kind of food is strictly prohibited during the Examination. If necessary, you may ask Lab Assistant and take a snack break nearby outside the Venue.
3. Participants are expected to work safely, to behave socially and to keep equipment and work environment clean. When carrying out discussions with your teammates, keep your voice low.
4. Do not leave the examination room until you have permission to do so. Ask Lab Assistant if you need to use the bathroom.
5. Work may only begin when the start signal is given.
6. You have 3 hours to complete the experimental tasks, and record your results on the answer sheets. There will be a pre-warning 30 minutes before the end of your time. You must stop your work immediately after the stop command is given. A delay in doing this by 5 minutes will lead to zero points for the task.
7. Be sure that each member of your team has a complete set of the experimental examination answer sheets (1 white copy for workout) and 1 yellow copy for submission). Submit only the yellow answer sheets.
8. Use only the pen and calculator provided.
9. Team code and student codes must be written on every page of the final answer sheets. Each team member must sign on the front page of the final answer sheets.
10. All results must be written in the designated boxes on the answer sheets. Data written elsewhere will not be graded.
11. After completing the task, put all the equipment back to its original place.
12. After the stop command is given, put ONLY the final answer sheets (one copy) on top of the envelope on the desk. Wait for the Lab Assistant to check and collect it. You can take the other papers with you.

EXAMINATION RULES

1. All competitors must be present at the front of examination room ten minutes before the examination starts.

2. No competitors are allowed to bring any tools except his/her personal medicine or any personal medical equipment.

3. Each competitor has to sit according to his or her designated desk.

4. Before the examination starts, each competitor has to check the stationary and any tools (pen, ruler, calculator) provided by the organizer.

5. Each competitor has to check the question and answer sheets. Raise your hand, if you find any missing sheets. Start after the bell.

6. During the examination, competitors are not allowed to leave the examination room except for emergency case and for that the examination supervisor will accompany them.

7. The competitors are not allowed to bother other competitor and disturb the examination. In case any assistance is needed, a competitor may raise his/her hand and the nearest supervisor will come to help.

8. There will be no question or discussion about the examination problems. The competitor must stay at their desk until the time allocated for the examination is over, although he/she has finished the examination earlier or does not want to continue working.

9. At the end of the examination time there will be a signal (the ringing of a bell). You are not allowed to write anything on the answer sheet, after the allocated time is over. All competitors must leave the room quietly. The question and answer sheets must be put neatly on your desk.

EXPERIMENT ONE: ESTIMATION OF GLUCOSE CONCENTRATION IN EXTRACTS OF LOCAL FRUITS

Introduction

The fruits of Date palm (*Phoenix dactylofera*), and Garden egg (*Solanum aethiopicum*) **(Plate 1)** are commonly consumed as snacks in Nigeria.
The date palm is fleshy with a sweet taste. It contains sugars, high amounts of fibre, vitamins, minerals and has a negligible fat content and may be consumed fresh or dried as a snack. The taste of garden eggs ranges from bland to sweet or even slightly bitter. It serves as a vegetable in stews and other sauces or may be consumed fresh. It is low in sodium, low in calories and very rich in dietary fibre.

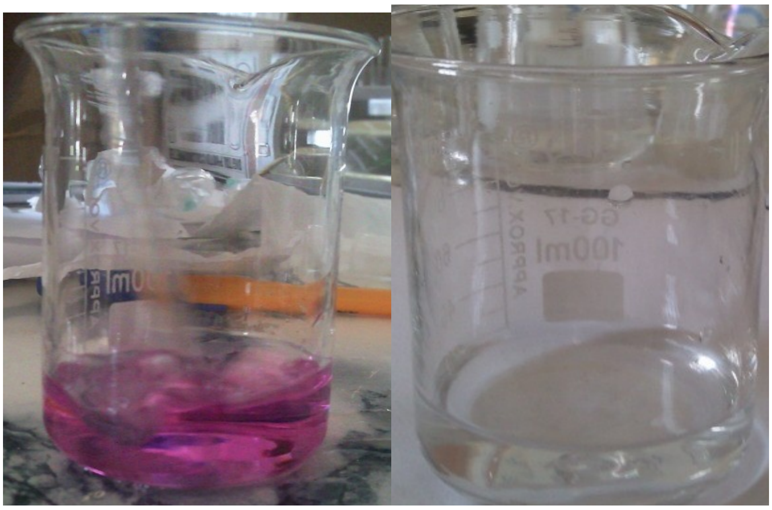
Solanum aethiopicum

Pheonix dactylofera

Plate 1: **Nigerian Tropical Fruits**

The aim of this experiment is to determine the concentration of glucose in the extracts of these fruits. You must determine the time it will take a variety of glucose solutions of known concentrations to decolourise solutions of potassium tetraoxomanganate (VII) ($KMnO_4$) and draw a Standard Curve from which the estimate of the glucose concentration in the extracts of local fruits will be determined. You need to measure the time taken for the purple colour of $KMnO_4$ to lose its colour completely.

Glucose ($C_6H_{12}O_6$) is a monosaccharide reducing sugar. In a chemical reaction with $KMnO_4$, the glucose molecules can cause the tetraoxomanganate(VII) (MnO_4^-) to lose its colour. The purple solution of MnO_4^- is reduced to a colourless solution of manganate ions (Mn^{2+}) as in **Plate 2**. The rate of decolourization of the MnO_4^- solution will be directly related to the concentration of glucose present in the mixture. Accuracy of timing and use of clean glassware and materials are some of the important factors that may affect the results of this experiment.



Purple colour ⟹ **Colourless**

Plate 2: Colour Change in Glucose, Tetraoxosulphate(VI) Acid and Potassium Tetraoxomanganate(VII) Reaction

Materials

1. Glucose solutions of known concentrations: G1 – G4 (Table 1)
2. Extracts from local fruits (A and B)
3. 1M solution of tetraoxosulphate (VI) acid
4. 0.01% w/v Potassium tetraoxomanganate (VII) solution
5. Conical flask (50 cm^3) with rubber corks (6)
6. Stop Watch (1)- 1 round is 30 seconds
7. 12 Syringes
   - (i) 10 cm^3(8)
   - (ii) 5 cm^3(2)
   - (iii) 2 cm^3 (2)
8. Marker (1)

Table 1: Glucose solutions of known concentrations provided.

Glucose solution	G1	G2	G3	G4
Concentration (%)	2.0	6.0	10.0	12.0

Experimental Procedure

1. Use the marker provided, to label four conical flasks G1 – G4 and place on the table serially.
2. Use separate 10 cm^3 syringes to transfer 10 cm^3 of each glucose solution into the labelled conical flasks as shown in Table 1.
3. Use a 5 cm^3 syringe to transfer 5 cm^3 of tetraoxosulphate(VI) acid into the conical flask labelled G1.
4. Use a 2 cm^3 syringe to transfer 2 cm^3 of potassium tetraoxomanganate(VII) solution into the conical flask G1 and **immediately** start the clock.
5. Shake the mixture in the conical flask G1 continuously and stop the clock as soon as purple colour disappears (**see Plate 2**).
6. Record in Table 2, the time taken for the purple colour to disappear completely.

Table 2: Glucose Concentration and Time Taken to Decolourise

Conical flask number	G1	G2	G3	G4
Glucose Concentration (%)	2.0	6.0	10.0	12.0
Time (mins)				

(2.0 marks)

7. **Repeat steps 3 – 6** for conical flask G2, G3 and G4 serially and complete Table 2 accordingly.
8. Use the marker to label two new conical flasks A and B, and place them on the table.
9. Use a separate 10 cm^3 syringe to transfer 10 cm^3 of extract A into conical flask A **and repeat steps 3-5** for the extract A.
10. **Record** in Table 3, the time taken for the purple colour to disappear completely.
11. Use another 10 cm^3 syringe to transfer 10 cm^3 of extract B into conical flask B and repeat steps **3-5** for the extract B.

12. Record in Table 3, the time taken for the purple colour to disappear completely.
13. In case you need to do experiment again, discard everything in the waste container and rinse the conical flasks with water before use again

Questions

1.1 Plot a graph of the results of G1 – G4 on the graph paper provided with time
on the Y (vertical) axis and the concentration of glucose on the X (horizontal) axis. **(2.0 marks)**

1.2 From the graph plotted, determine the concentration of glucose in samples A and B. **(2.0 marks)**

Table 3: Time Taken to Decolourise Test Solutions (2.0 marks)

Conical flask	A	B
Glucose conc. (%)		
Time (mins)		

1.3 Which of the samples A or B has the higher concentration of glucose? **(1.0 mark)**

1.4 Glucose is considered a reducing agent in this experiment because; (Tick the appropriate boxes below)

Option	Reason	True	False
i	oxidation number of Mn is decreased		
ii	oxidation number of Mn in MnO_4^- became +4		

(1.0 mark)

1.5 Complete the following paragraph by using the most appropriate letters from the key below. **(2.0 marks)**

During the process of photosynthesis green plants use _______ gas to synthesise glucose. This process occurs in light in the organelle called _______. An inorganic substance, ___________, is also a reactant in the process. The glucose that is manufactured is stored mainly as ________ in the plants. The glucose in the fruits plays a role in the dispersal of the seeds. Animals are attracted by the _______ of the fruit and they eat it. The seeds have a hard________ which prevents the seed from being

_______ by the ________ in the alimentary canal of the animals. Later the animal ________ the seeds, usually away from the parent plant. This helps reduce ___________ between the parent plant and its offspring.

A – Mitochondria	K – Oxygen
B – Seed Coat	L – Assimilates
C – Carbon Dioxide	M – Competition
D- Water	N – Starch
E – Mucus	O – Magnesium
F – Chloroplasts	P – Egests
G – Enzymes	Q – Colour
H– Vacuole	R – Variation
I -Endosperm	S – Texture
J – Digested	T – Glycogen

1.6 In a similar experiment two fruits C and D were investigated for glucose concentration. Fruit C had a higher concentration of glucose. Jauro Amadu (JA) had been diagnosed to have non-functional beta cells of the Islets of Langerhans. If JA must eat the fruit C or D, which of the samples C or D would you preferably recommend for his consumption? **(1.0 mark)**

Answer ____

1.7 Give reason for your answer in **1.6** by ticking True or False in the boxes below. **(1.0 marks)**

Options		True	False
i	JA does not produce insulin		
ii	Fruit C has more water content than fruit D		
iii	Fruit C contains more glucose		
iv	JA does not produce glucagon		

EXPERIMENT TWO: RENEWABLE ENERGY SOURCES

Introduction

Nigeria is one of the world's leading petroleum producing nations. Petroleum is the major foreign exchange earner for Nigeria. However, like most forms of non-renewable energy petroleum is exhaustible. There is therefore a new impetus to look for alternative sources of energy that will be sustainable.

Biodiesel happens to be one of such sources of energy which is obtained mainly from plant and animal fats. It has similar applications to petro-diesel and can be used without modification of the engine. Biodiesel when compared to petro-diesel releases less pollutants into the atmosphere during combustion. Since it is obtained from plant and animal fats, it is regarded as a renewable energy source.

Fuel quality characteristics for diesel fuels include viscosity, flash point, cloud point, pour point and acid value.

Equations (1) and (2) can be rearranged to obtain expressions for absolute viscosity:

$$8lV\eta = \pi gh\rho r_0^4 \Delta t \quad \text{.....................} \quad (1)$$

$$8klV = \pi ghr_0^4 \quad \text{..........................} \quad (2)$$

Where l = length, π= constant, g: acceleration due to gravity, h= height of viscometer, ρ = density of liquid, r_o= radius of the tube, Δt = time for test liquid to pass through two points, η = absolute viscosity, V= total volume run out for the total time Δt , and k = parameter which will be constant if Δt and ρ are constant.

Objectives

In this task you will be required to prepare biodiesel from Palm Kernel Oil (PKO) obtained from the palm tree (*Elias guinesis*) which is very abundant in Nigeria.

1. Preparation of biodiesel from PKO
2. Estimation of the percentage mass/mass yield of biodiesel from PKO
3. Determination of acid value of PKO-biodiesel and PKO itself.

Apparatus / Materials

a) Palm Kernel Oil (PKO) (100 cm^3) (density 0.912 g cm^{-3})

b) Methanol(50 cm^3) **NOTE Methanol is a safety hazard so avoid inhalation and wear safety goggles when handling**.

c) Potassium hydroxide (KOH) (30 pellets)

d) Cotton wool

e) Water(500cm^3)

f) Anhydrous magnesium sulphate ($MgSO_4$) (1 sachet)

g) 250 cm^3 quick-fit flat bottomed round flask(1)

h) 250cm^3 conical flasks(4)

i) 250 cm^3 beakers(4)

j) Wash bottle(1)

k) 100 cm^3 measuring cylinder(2)

l) 125 cm^3 separating funnel (1)

m) Spoon spatula(1)

n) Glass funnel (1)

o) Burette (1)

p) 0.01 mol dm^{-3} (or mol l^{-1}) Potassium Hydroxide solution (a stock solution)

q) Phenolphthalein Indicator

r) Ethanol C_2H_5OH

s) Stop watch (1)

t) Stirring rod (1)

u) Pipette and filler

Procedure for the Preparation of Biodiesel

1. Using a spatula, place 5 pellets of potassium hydroxide (KOH) into a dry 250 cm^3 quick-fit flat bottomed round flask and stopper the flask.

2. Use the measuring cylinder to transfer 10 cm^3 of methanol (CH_3OH) into the flask. Seal with the stopper and shake vigorously until the potassium hydroxide (KOH) dissolves. NOTE: This process is exothermic so take care with the stopper

3. With the measuring cylinder transfer 30 cm^3 of PKO to the flat bottomed round flask, seal with the stopper and shake the mixture vigorously for 15 minutes,

4. Empty the entire contents of the flat bottomed round flask into the 125 cm^3 separating funnel and allow the mixture to stand for approximately 7 minutes, without a stopper. Run off the bottom layer into a beaker. The top layer is the crude biodiesel.

5. Use the measuring cylinder to transfer 40 cm^3 of water to the crude biodiesel in the separating funnel, swirl the funnel slowly without the stopper but ensure no liquid is spilt. Allow it to separate into two layers and run off the bottom layer into the beaker. Repeat this step twice to wash the biodiesel. Collect the bottom layer in the same beaker.

6. Run off the biodiesel into a 250 cm^3 beaker and slowly empty the sachet of anhydrous magnesium sulphate ($MgSO_4$) into the biodiesel.

7. Stir the mixture slowly with a stirring rod, allow it to settle for 60 seconds and slowly decant the biodiesel into a clean measuring cylinder. Filter any remaining biodiesel using the funnel bearing a tiny plug of cotton wool into the same measuring cylinder.

8. Record the volume of biodiesel produced.

Determination of Acid Value of PKO-Biodiesel

1. Place 2.0 cm^3 of the biodiesel using the pipette and filler, into a conical flask

2. Add ethanol (10 cm^3) to the conical flask and shake for 60 seconds

3. Pour 0.01 mol dm^{-3} (mol l^{-1}) Potassium Hydroxide (KOH) stock solution into the burette

4. Titrate the biodiesel solution against the 0.01 mol dm^{-3} (mol l^{-1}) Potassium Hydroxide (KOH) stock solution using phenolphthalein indicator
5. Record the titre value.
6. Repeat the titration at least once again.

Determination of Acid Value of PKO

1. Place 2.0 cm^3 of the PKO using the pipette and filler, into a conical flask
2. Add ethanol (10 cm^3) to the conical flask and shake for 60 seconds
3. Pour 0.01 mol dm^{-3} (mol l^{-1}) Potassium Hydroxide (KOH) stock solution into the burette
4. Titrate the PKO solution against the 0.01 mol dm^{-3} (mol l^{-1}) Potassium Hydroxide (KOH) stock solution using phenolphthalein indicator
5. Record the titre value.
6. Repeat the titration at least once again.

QUESTIONS

2.1. From the list given, choose two substances that constitute the bottom layer obtained in step 4 from the preparation of the biodiesel:

(i) Potassium Hydroxide (KOH)
(ii) Water
(iii) PKO
(iv) Biodiesel.

(0.5 x 2 = 1.0marks)

2.2 Calculate the percentage yield by mass of PKO-biodiesel from PKO based on your results. (Assume PKO-biodiesel density is 0.89 g cm^{-3}) **(2.5 marks)**

2.3 Why is magnesium sulphate added in step 6 in the extraction of PKO-biodiesel? Select the correct option from the table below **(0.5 mark)**

Option	Reason
A	To improve the conductivity
B	To reduce the oil to hydrocarbons
C	To remove any remaining water
D	To increase the viscosity of the biodiesel

2.4 From equations 1 and 2 derive the expression for absolute viscosity η **(1.0 mark)**

2.5 Record the titre value you obtained in the acid determination of PKO **(1.5 mark)**

2.6 Using the formula Acid value = (V x c x Z)/ m, calculate the acid value.

Where V= volume in dm^3 / l of 0.01 mol dm^{-3} (mol l^{-1}) Potassium Hydroxide (KOH) solution consumed (titre value)

c = concentration of Potassium hydroxide (KOH) solution
m = mass (g) of PKO sample
Z = 56.1 g/mol

Ensure you use the appropriate units and assume 1 cm^3 of PKO weighs 0.912 g

(1mark)

2.7 Calculate the acid concentration in mol dm^{-3} of PKO.(K = 39.1, O = 16.0, H = 1.0 **(1 mark)**

2.8 Record the titre value you obtained in the acid determination of PKO-biodiesel **(1.5 marks)**

2.9 Using the formula Acid value = (V x c x Z)/ m, calculate the Acid value of PKO-biodiesel.

Where V= volume in dm^3 / l of 0.01 mol dm^{-3} (mol l^{-1}) Potassium Hydroxide (KOH) solution consumed (titre value)

c = concentration of Potassium hydroxide (KOH) solution
m = mass (g) of PKO-biodiesel sample
Z = 56.1 g/mol

Ensure you use the appropriate units and assume 1 cm^3 of PKO-biodiesel weighs 0.89 g **(1 mark)**

2.10 Calculate the acid concentration in mol dm^{-3} of the PKO-Biodiesel. (K = 39.1, O = 16.0, H = 1.0) **(1 mark)**

2.11 Provide the correct option from A-D to account for the differences in the observed acidity of PKO and PKO-biodiesel **(0.5 mark)**

A. Method of preparation of PKO- Biodiesel makes it more volatile
B. Magnesium sulphate was used in the extraction of PKO-Biodiesel
C. In the extraction PKO-biodiesel it was mixed with potassium hydroxide (KOH) which neutralized the acidity
D. The extraction process increases the yield of PKO-Biodiesel

2.12 Select the best option in the list below for the reason that biodiesel releases less pollutants into the atmosphere than petro-diesel when combusted. **(0.5 mark)**

A. It contains more oxygen

B. Biodiesel contains less sulphur.

C. It contains more carbon atoms

D. It is highly dense

EXPERIMENT THREE: MEASUREMENT OF THE VISCOSITY OF CASTOR (*ricinus communis*) OIL

3.1 Introduction

It is known that as a spherical metal ball of radius, r and density, ρ_s falls under gravity through a viscous liquid of density, ρ_l, the ball experiences opposing forces down to a point where the dynamics of the motion is given as:

$$\frac{4}{3}\pi r^3 \rho_\ell g + 6\pi r \eta_\ell v_o = \frac{4}{3}\pi r^3 \rho_s g \qquad (1)$$

where g is the acceleration due to gravity and η_ℓ is the coefficient of viscosity of the liquid and v_o is the velocity of the spherical ball at equilibrium of the forces, (i.e. terminal velocity).

3.2 Objective

The objective of this experiment is to determine the coefficient of viscosity of castor oil using equation (1).

3.3 Apparatus

The apparatus has already been set up (see Fig. 1) and basically consists of a long graduated cylinder filled with castor oil.

You are also provided with forty (40) spherical metal balls, each of diameter 4.76 mm and two stop watches.

3.4 Procedure

3.4.1 Study the apparatus as set-up in Figure 1(you may call the attention of the invigilators if the placement of the apparatus is inconvenient, for example the vertical position of the cylinder. DO NOT MOVE THE APPARATUS BY YOURSELF).

3.4.2 Pick one of the spherical metal balls provided and carefully drop it as close as possible to the liquid surface in the glass cylinder and approximately at the centre.

3.4.3 Using the stop watches provided, time the motion of the ball as it falls through lengths of the liquid column as given in Table 1, taking the 20 cm mark as the starting point of timing. **If the ball touches the wall of the cylinder, call the attention of the invigilator.**

3.4.4 Record the time (t_1) taken for the ball to fall from the starting point to the 40, 50, 60, 70, 80, 90, 100 and 110 cm marks on the glass cylinder.

3.4.5 Repeat step 3.4.4 and record the time (t_2).

3.4.6 Determine the average of t_1 and t_2 and record it as t.

Fig. 1: Experimental set-up

Table 1: Table of values

Distance marks on the tube (cm)	Distance travelled (cm)	Time (s)		
		t_1	t_2	t
20	-	0.00	0.00	0.00
40				
50				
60				
70				
80				
90				
100				
110				

(3.2 marks)

3.5 Questions

3.5.1 Plot the graph of distance travelled against time. **(1.6 marks)**

3.5.2 Determine the slope of the graph in question 3.5.1 **(1.2 marks)**

3.5.3 Identify the physical meaning of each of the three (3) terms in equation (1) labeled A, B, and C in Table 2 using Table 3 which gives the likely names of the terms (i.e, match Table 2 and Table 3 using the appropriate Table in the answer booklet). **(1.5 marks)**

Table 2

A	B	C
$\frac{4}{3}\pi r^3 \rho_\ell g$	$6\pi r \eta_\ell v_o$	$\frac{4}{3}\pi r^3 \rho_s g$

Table 3

I	II	III	IV	V
Gravitational force (weight)	Strong force	Buoyant force (upthrust)	Viscous force	Centrifugal force

3.5.4 Rearrange equation (1) to make η_ℓ the subject of the equation. Call this equation (2). **(1.2 marks)**

3.5.5 Given that v_o is the slope determined in question (3.5.2) and that g = 9.8 ms^{-2}, ρ_ℓ = 900 kgm^{-3}, ρ_s = 7800 kgm^{-3}, calculate η_ℓ for the castor oil using equation (2).

(2.3 marks)

3.5.6 The factors in Table 4 may affect the value of the coefficient of viscosity measured at different locations on the earth's surface by this method. Tick (√) as appropriate

(1.0 mark)

Table 4

	True	False
Altitude		
Latitude		
Relative humidity		
Ambient temperature		

3.5.7 Precautions which may be taken in order to obtain a precise result are as in Table 5. Tick (√) as appropriate **(1.0 mark)**

Table 5

	True	False
Minimize parallax error		
Avoid the balls touching the walls of the glass cylinder		
Changing the starting point of timing to 50 cm		
Dropping the ball from a height above the liquid surface		