

Qualitative Analysis of Oil Palm (*Elaeis Guineensis*) Bunch Refuse Ashes

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Abstract - The qualitative analysis carried out on the *Elaeis guineensis* fruit Bunch refuse ashes showed the presence of nitrogen, sulphur and chlorine as the major elemental constituents. The study also proves that the material contains Nitrogenous basic compounds. Thus primary aromatic amine is very prominent as the material undergoes diazotization and coupling reaction to form dye. Also the above assertions were based on the test, observation and deductions derived from literature and laboratory work which include Alkalinity test, solubility test, elemental and functional group analysis.

Keywords: Diazotisation, coupling, sulphonamide, alkaloid, fusion.

1. INTRODUCTION

The oil palm which produces the bunch refuse belongs to the family “palmae” which is known as “*Elaeis guineensis*” in Africa and *Elaeis oleifera*” in South America as its scientific names, locally, it is known by different names in various parts of Nigeria such as “Kwakwa” by Hausas “Zukunnu” by Nupe, “irile” by Tiv, “Ope” by Yoruba’s “Akpuud – eyop by Ibibios. Others are udin, orien and Nkwu by Binis, Urhobo and Igbos respectively.

Oil palm is native to West Africa but it is cultivated today in Malaysia, Sumatra, Central America, Venezuela, and Brazil (Obahiagbon, 2012). It is a monocot that has an unbranched habit which produces a single trunk that bear a crown of fronds. The tree often attains a height of 60 feet and as monoecious plant, it produces two types of inflorescence separately on the same tree (Corley and Lee, 1992).

The female inflorescence is the major raw material for this research work, arises from a stout but shorter peduncle supporting about 150 branches, spikes forming together an ovate compact inflorescence and each branch bearing about 12 flowers. The fruits developing from the flowers are drupes from the fleshy mesocarp. This fleshy mesocarp yields the palm oil while the endospermous seed of the drupe is the source of palm kernel (seed) oil. Four different variations of fruits are known. They include the group “macrocarp” which is the variety that produces fruits with very thick stony endocarp. The group “Dura” has a hard endocarp of intermediate thickness and the group “Tenera” produces drupes with a very thin endocarp while the variety that does not form any visible endocarp is called “Pisifera” (Paterson, 2007).

Also, oil palm requires an average temperature of 22°C – 23°C. Average daily sunshine should be up to 6 hours per day and

rainfall of about 2,000mm (80 inches) or more per annum (AIST, 2006) which is every distributed throughout the year.

The soil must be very rich in Nitrogen, potassium and magnesium. Deficiency of any of these causes a great reduction in the yield of fruits and affects the growth of the bunch (Clay, 2004).

The economic value of the oil palm is enormous thus; its leaf – ribs are used in building, the leaves in thatching, the fibre in rope making. The palm wine obtained by tapping the tree is a pleasant intoxicating drink, the palm oil is a valuable source of vitamins in the indigenous diet (Fargione *et al.*, 2008) as well as palm kernel cake, a major ingredient in livestock feed. (Fargione *et al.*, 2008).

The palm oil (pericarp oil) and the kernel oil obtain from palm fruits which are joint products of the palm bunch refuse under these two broad areas, many researches have been done and as a result, maximum use of these products has been ensured. To this end, an insight into the value of the palm bunch refuse is expected. Though it is used locally in soap making by acting as saponifying agent, its contribution to the economic development of the nation is still obscure since the resources has not been tapped to its full utilization. Hence this research is aim at finding its hidden qualities and therefore it’s utility towards the Nation’s economy.

MATERIALS AND METHODS

2.1 Processing of Raw Material

The raw material (oil palm bunch refuse) was obtained from forest of Ntak-Inyang Itam, a village in Itu Local Government Area of Akwa Ibom State. Ten fresh matured bunches were cut from oil palm tree and kept for 6-days for the fruits to loosen. This was followed by drying of the bunch refuse under direct sunlight for seven days (one week) after which the bunch refuses were burnt together in a clean pan. The ash which has a combination of grey and black colour was pulverized in a Porcelain Mortar to obtain homogeneous mixture of the powder. This was further dried and stored in a plastic container for subsequent analysis.

2.2 Extraction of the Organic Constituents

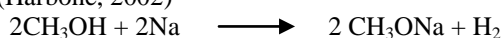
The extraction was performed with diethyl ether and ethanol according to method described by Harbone (2002). 20g each of the bunch refuse ash were dissolved in 50ml of distilled H₂O in two different reagent bottles. The solution were emptied into different separation bottles and 50mls each of the

two solvent were added into the separate separation bottles in portions of 20ml, 15ml, 10ml.

Each portion was allowed to settle for 30 minutes in the separating funnel after thorough shaking, so that proper separation of the organic layer from the aqueous layer was achieved. The extracts were filtered and concentrated using water bath. These were labeled ether and ethanol extract respectively.

Elemental Analysis

A small piece of sodium of about 3mm in diameter was taken in a fusion tube and was heated to redness by a direct flame. With the help of thong, the tube was held vertically and 10mg (2 drops of extract) of the substance was dropped directly onto the red hot sodium to avoid explosion. Heating was continued for about 2 minutes at which time the bottom of the tube became red. The tube was then allowed to cool and about 5 drops of methanol were added to neutralize the excess sodium (Harbone, 2002)



The fusion tube was crushed in a mortar to allow for maximum contact between the methanol and excess of sodium. 2ml of distilled water was then added to dissolve the fusion product. The solution was filtered and divided into portions.

Detection of Nitrogen

Freshly prepared saturated solution of FeSO_4 was added to the first portion of the filtrate. It was heated for 30 seconds at 100°C using water bath, 2 drops of ferric sulphate solution were added. The solution was then acidified with H_2SO_4 . The above procedure was repeated with the solvent extract and a blank test was also performed without the substance.

Detection of Sulphur

- The second portion (23ml) of the filtrate was added to a test tube containing 0.1g of powdered FeSO_4 crystals.
- 2ml of the fusion solution was acidified with dilute acetic acid and a few drops of lead acetate solution were added. These two tests were also performed with the solvent extracts including the blank test.

Detection of Halogens

The third portion of the filtrate was acidified with 2M HNO_3 . The solution was heated at 100°C to reduce the volume to about half. After cooling, 3ml of aqueous solution of AgNO_3 was added. A white precipitate was observed.

Another portion of the fusion solution was acidified with 2M HCl . 1cm³ of CCl_4 and a 3 drops of chlorine water were added. The mixture was shaken and separation and colour of the CCl_4 layer was noted in the separating funnel. The solvent extract was also used to run the same experiment. The blank test was equally done.

Classification Test

Acidity and Alkalinity Test

The aqueous solution of the sample was tested with litmus paper and phenolphthalein indicator. Also about 0.2g of the sample was dissolved in ethanol and a drop of

phenolphthalein added. Then NaOH solution was added drop wise to obtain a permanent pink colour.

Solubility Test

0.1g of the sample was placed in a test tube and 3ml of water was added in portion of 1 ml with vigorous shaking. For the solubility of the substance in dilute NaOH, NaHCO_3 and HCl solutions, the mixture was thoroughly shaken and filtered. The filtrate were neutralized with HCl, in the case of NaOH and NaHCO_3 as solvent while NaOH was used in the case of HCl as solvent (Robert, 2005).

Functional Group Test

Nitrous Acid Test for Amine

0.1g of the substance was dissolve in 3ml of concentrated HCl diluted with 5ml of distilled H_2O . The solution was cooled to 0°C in a beaker containing cracked ice.

Similarly 1g of sodium nitrate was dissolved in 5ml of water and was added slowly with shaking to the cold solution of the amine hydrochloride in the first test tube. The addition of NaNO_2 solution was continued until the mixture gave a blue colour on starch iodide paper (positive test for nitrous acid) as described by Robert (2005). The solution was divided into two:

- To the first portion of the cold diazonium solution in a test tube, a solution of 0.1g 1-naphthol in 2ml of 10% NaOH solution and 5ml of water was added.
- The second portion of the cold diazonium solution which showed positive test for nitrous acid was warmed with water bath. The bubbling action and colour changes where accurately noticed. Blank test was also carried out.

TEST FOR HALOGEN COMPOUNDS

Alcoholic Silver Nitrate Test

0.2g of the ash was added to 2ml of 0.05M ethanolic AgNO_3 solution. The white precipitate which forms was dissolve in dilute HNO_3 and NH_4OH to identify the halogen precipitated (Robert, 2005).

Test for Alkaloid

- Extraction: This was done using 2 polar solvents-water and 10% H_2SO_4 . 5g each of the powdered as was heated differently with the solvent for about 10 minutes. The mixtures were filtered and tested as follows:
- Test: This was carried out by adding 3 drops of Wagner's reagent to 1ml of alkaloid solution. A blank test was also carried out and all the colour changes were noted.

RESULT AND DISCUSSION

Result

The results of this research work is tabulated under the following readings; test, observation, and inference.

Elemental Analysis

Test	Observation	Inference
Nitrogen		
i. Fusion solution + FeSO ₄	Greenish gelatinous precipitate form	Iron(II) continuous or salt present
ii. Fusion solution + FeSO ₄ + Fe ₂ (SO ₄) ₃ + H ₂ SO ₄	The gelatinous precipitate dissolved giving a greenish blue solution	Nitrogen is present
iii. Sodium solution + FeSO ₄ + Fe ₂ (SO ₄) ₃ + H ₂ SO ₄	Yellow coloured solution observed	Nitrogen is not present
Sulphur		
iv. 2ml of fusion solution + 0.1g FeSO ₄ crystal	Black precipitate observed	Sulphur is present
v. 2ml of sodium solution + 0.1g FeSO ₄ crystal	Greenish gelatinous precipitate formed	Sulphur is not present
vi. 2ml of fusion solution + CH ₃ COOH + Pb(CH ₃ COO) ₂	Black precipitate was formed	Presence of sulphur confirmed
vii. 2ml of sodium + CH ₃ COOH + Pb(CH ₃ COO) ₂	No change in colour	Sulphur is not present
Hallogen		
viii. 2ml of fusion solution + 2M HNO ₃ + AgNO ₃	White precipitate was obtained	Cl, Br, Or I is present
ix. White precipitate + (viii) + HNO ₃	Insoluble	Cl, Br or I is present
x. 2ml of sodium solution + 2M HNO ₃ + AgNO ₃	No precipitate	Cl, Br, or I present
xi. 2ml of fusion solution + 1 ml CCl ₄ + 3 drops of chlorine water + shaking	Solution separate into two. No change in colour was observed in the CCl ₄ layer	Chlorine is suspected
xii. White precipitate in (viii) + NH ₄ OH	White precipitate dissolved	Chlorine (Cl) confirmed
Basicity/Acidity Test		
xiii. Aqueous solution of the substance + litmus	Litmus paper turn blue	Substance is basic
xiv. 0.2g of the substance in ethanol + 1 drop of phenolphthalein	Pink colour was observed without the addition of NaOH.	Substance is basic
xv. 0.1g of	Insoluble. No	The substance

substance + 3ml of 5% NaHCO ₃	effervescence	acidic
Solubility Test		
xvi. 0.1g of substance + 3ml of water	A homogenous black solution obtained	Substance is soluble. Salt and amines suspected
xvii. 0.1g of substance + 3ml of ether	Substance remained in its solid state and no change in colour was obtained	Substance is insoluble ionic compound (salt) suspected
xviii. 0.1g of substance + 3ml of NaOH	No conspicuous change after shaking	
xix. Filtrate of (xviii) + 5% HCl	No separation or cloudy solution was observed	Substance insoluble basic substances are insoluble in NaOH
xx. 0.1g of substance + 3ml of 5% NaHCO ₃	No change was observed	
xxi. Filtrate of (xx) + HCl	No separation of cloudy solution was observed	Substance is insoluble basic do not dissolve in NaHCO ₃
xxii. 0.1g of substance + 3ml of HCl	Effervescence was observed. Test tube became hot	
xxiii. Filtrate of (xxii) + NaOH	White cloudy solution was formed	Substance is soluble nitrogenous bases are present. Primary amine suspected since secondary and tertiary amines are insoluble
Amine		
xxiv. Amine hydrochloride solution + 3 drops of NaNO ₂ solution + starch iodide paper at 0°C	Starch iodide paper turn blue showing the presence of excess HNO ₃ acid	Diazonium salt is formed
xxv. Diazonium solution + 0.1g 1-naphthol in 2ml of 54% NaOH and 5ml of water	Deeply brown coloured precipitate (dye) was obtained	Aromatic amine is present (Vogel <i>et al.</i> , 1989)
xxvi. Cold diazonium – solution + heat	Bubbling was observed due to evolution of	The aromatic amine is primary

	Nitrogen. Evolution of brownish gas of oxides of Nitrogen was also noticed	(Vogel <i>et al.</i> , 1989).
Hallogen Compound		
xxvii. 0.2g of the substance + 2ml of 0.05m ethanolic AgNO ₃ solution	White precipitate formed	Chlorine compound present
xxviii. White precipitate in (xxvii) + HNO ₃	Insoluble	Chlorine compound present
xxix. White precipitate in (xxvii) + NH ₄ OH	Soluble	Chlorine compound is confirmed present

Alkaloid		
xxx. 1 ml of aqueous alkaloid + 3 drops of Wagners reagent	Colour of the reagent persist no reddish brown precipitate	Alkaloid not present
xxxi. 1ml of water + 3 drops of Wanger's reagent	Colour of the reagent persist	Alkaloid not present
xxxii. 1ml of Alkaloid sulphate + 3 drops of Wagners reagent	No reddish brown precipitate	Alkaloid is not present
xxxiii. 1 ml of H ₂ SO ₄ + 3 drops of Wagner's reagent	No reddish brown precipitate was observed	Alkaloid is not present

DISCUSSION

The elemental analysis has given the evidence that nitrogen, sulphur and chlorine are present in the sample. The solubility test has equally shown that, the substance contains polar ionic compounds such as salt since it dissolves in water. Similarly, the insolubility in ether suggests that the material is made up of strongly polar ionic compounds such as salt. This is because it is well established that if a substance dissolves in water but not in ether, it may likely be ionic (salt) (Vogel, *et al.*, 1989).

As regards, the Alkalinity/Acidity test, it was deduced that the substance is a base having tested positive to litmus paper and phenolphthalein. The insolubility of the substance in 5% NaOH and 5% NaHCO₃ has ruled out the presence of Acidic substance while it's solubility in 5% HCl is in line with the basic nature of the substance. It therefore classifies the material as a nitrogenous base, it also suggest that aliphatic

and primary aromatic amines are most likely present. Secondary and tertiary aromatic amines such as diphenyl amine and triphenyl amines are excluded because they are insoluble in HCl due to the fact that the basic character of the nitrogen atom has been diminished to such an extent that they do not form salt with dilute HCl (Robert, 2005).

Under functional group analysis, the material tested positive to nitrous acid test for amine. It undergoes diazotization and couples with 1-naphthol to form dye (Vogel *et al.*, 1989). This result further classified it as an aromatic amine. Furthermore, the diazonium solution formed with the substance was found to give off nitrogen gas on heating, thus suggesting that the material contains a primary aromatic amine.

In addition, the positive test of the material to Alcoholic AgNO₃ test for Halogens compounds confirms the presence of chlorine and suggest among other compound the following salts.

- i. RCOO⁻M⁺ – salt of fatty acid
- ii. RSO₃⁻M⁺ – salt of sulphoric acid
- iii. RNH₃⁺Cl⁻ – salt of primary amine

This assertion is in agreement with the inference drawn under liability test. However, the possibility of the polar ionic (salt) compound being RCOO⁻M⁺ and RSO₃⁻M⁺ is rule out since the material tested negative towards acidity. So far, the presence of RNH₃Cl⁻ has been favoured based on the observation made where R may be a phenyl or phenyl alkyl radical.

CONCLUSION

The elements nitrogen, chloride sulphur and aromatic primary amine have been confirmed present and are all supported with cogent reason. However, it was not easy to determine a compound of sulphur in the material, the ones suspected present such as sulphanamide has its properties not in agreement with the general properties of the material.

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