ABUAD Journal of Engineering Research and Development (AJERD) ISSN: 2645-2685
Volume 2, Issue 2, 25-31

Extraction of Palmitic Acid from Plantain (*Musa paradisiaca*) Stalk Residue

Adewale George ADENIYI[1], Kevin Shegun OTOIKHIAN[2], Damilola Victoria ONIFADE[1], Joshua O. IGHALO[1], Mustapha A RAJI[2], Samuel Oluwasegun OLONADE[1]

[1]*Department of Chemical Engineering University of Ilorin, P. M. B. 1515, Nigeria*
adeniyi.ag@unilorin.edu.ng /damilola.onifade4@gmail.com /oshea.ighalo@yahoo.com / samuelnade4lyf@gmail.com

[2]*Chemical Engineering Department, Faculty of Engineering, Edo University, Iyamho, Nigeria*
otoikhian.kevin@edouniversity.edu.ng

[3]*Department of Industrial Chemistry, University of Ilorin, P. M. B. 1515, Nigeria*
mustaphaadekunle48@gmail.com

Corresponding Author: adeniyi.ag@unilorin.edu.ng

Date Submitted: 01/03/2019
Date Accepted: 15/08/2019
Date Published: 31/12/2019

Abstract: *This work is aimed at selective solvent extraction of palmitic acid from Plantain Stalk Residues (PSR). Plantain stalk obtained from the waste stream of a local market was used in this study. The stalk was grated and the extract obtained during the grating process was oven dried at a temperature of 70°C. The residue referred to as NT was characterized using Gas chromatography–Mass Spectrometry (GCMS) to ascertain the presence of fatty acids with much preference given to palmitic acid. This residue was dissolved in four solvents (water, ethanol, hexane and acetone) and also oven dried at 70°C. The dried residues were analyzed for their functional groups and inorganic content using studied using Fourier Transform Infrared FTIR, and X-Ray Fluorescence (XRF). These characterizations were conducted to determine the suitable solvent for a greater yield of palmitic acid. GCMS result* shows that *the percentage of palmitic acid expressed as percentage fraction of total fatty acids present in the plantain stalk was 14.4%. FTIR analysis results gave acetone as the most suitable solvent in obtaining a better yield of palmitic acid from PSR. Palmitic acid extracted from plantain stalk residue is suitable for applications in pharmaceuticals, food additives and soap making due to the high composition of Calcium, Silicon and Potassium respectively*.

Keywords: *Palmitic acid, plantain, extraction, acetone, hexane, ethanol, water*

1. INTRODUCTION

The modern society has seen a huge growth in the research and development of drugs whose raw materials are obtained from plants (fruits, leaves, roots and stems). Although some drugs are developed by synthesizing the known chemical structures of plant extracts through laboratory methods, the high cost of synthesis and the side effects still make the plant chemical a more preferable option [1, 2].

Plantain is one of the principal food resources in the world and its trees are grown in large quantities in tropical and subtropical countries. Significant quantities of plantain stalk are generated which causes nuisance to the environment making it essential to find applications for these stalks. Potential applications of plantain stalk depend on its chemical compositions.

Palmitic acid also known as hexadecanoic acid or palmitate with a molecular formula $C_{16}H_{32}O_2$ is a saturated non-polar fatty acid (lipid) found naturally in palm oil, olive oil, cheese, and body lipids [3, 4]. It is also present in butter, cheese, milk and meat, but it generally comprises less than 5% of the total fatty acid content in these sources (although this is much higher than the concentration of myristic acid and stearic acid). It could take the form of a liquid, solid (crystals) dry powder with color ranging from colorless to white. Palmitic acid is used to produce cosmetics, soaps, industrial mould release agents and also used in the determination of water hardness. Applications of palmitic acid can be found in the health and beauty (e.g. cosmetics, soap production), food (e.g. additive, texturing agent), pharmaceutical (e.g. palmate ester as a carrier medium and releasing agent) and biotechnology industries. In the health sector, palmitic acid has also been proven to mediate hypothalamic insulin resistance [5, 6]

The separation and purification of saturated fatty acids mixtures is carried out by crystallization, which makes solubility in solvents an important feat in designing and controlling the process [3, 7]. Palmitic acid is a non-polar compound which implies that a non-polar solvent would likely be more effective for its dissolution. The solubility of palmitic acid in several organic solvents has been studied by several researchers [4, 6, 8-11]The

commonly used organic solvents for lipid extraction include methanol, petroleum ether, isopropanol, diethyl ether, n butanol, chloroform, ethanol, acetone, isopropyl ether, acetonitrile, tetrahydrofuran, dioxane, pentane, dichloromethane, benzene, hexane, iso-octanol, cyclohexane, or mixtures of these solvents Fatty acids are insoluble in polar solvents like ambient water but highly soluble in weak polar or nonpolar organic solvents[12].

Researchers have dealt with several applications of plantain fiber but no studies on the extraction of palmitic acid from plantain fiber were found in literature. Natural fibers such as plantain has been found to be important in the reinforcement of polymer to for natural fiber polymer composites. Cadena and his colleagues investigated the properties of mechanically extracted plantain fiber for use in the reinforcement of polyester composites [13]. Imoisili *et al.* [14] also investigated the effect of chemical treatment on the mechanical and morphological properties of plantain fiber.

This research is aimed at extraction of palmitic acid from plantain stalk residue and selecting the most suitable solvent for the extraction.

2. MATERIALS AND METHODS

2.1 Collection and Preparation of the stalk samples.

The plantain stalks were collected from the waste stream of Ganmo Market in the metropolis of Ilorin. The cleaned stalks were cut into smaller discs after which they were pounded using a mortar and a pestle so as to remove the fibers contained within the stalk. After the removal of the fibres (Figure 1), a residual solution remained in the mortar. This solution was allowed to settle for 24hours. The filtrate solution was decanted manually and the residue left was collected and dried in the oven at a temperature at 70°C. The residue is referred to as NT and it was analyzed using Gas chromatography–Mass Spectrometry (GCMS), Fourier Transform Infrared FTIR, and X-Ray Fluorescence (XRF).

Figure 1: Extracted fibers

2.2 Dissolution of Sample and Characterization

NT was divided into five parts of which four are dissolved in Acetone, Ethanol, Hexane and Water respectively in order to selectively concentrate Hexadecanoic acid. The four mixtures were then placed in a constant temperature water bath and agitated till a uniform solution was obtained. Each of these solutions were then filtered using with a filter paper (Figure 2) and the residue was washed with distilled water and afterwards re-filtered and oven-dried. The residues obtained were thereafter collected together with NT for GCMS, FTIR and XRF analysis and characterization and the four dissolved extracts were sieved and dried in the oven after which they were analyzed for the organic components, functional group and inorganic components using GCMS, FTIR and XRF respectively.

Figure 2: Filtered Residues obtained from Acetone, Hexane and Ethanol Solutions

3. RESULTS AND DISCUSSION

3.1 Gas Chromatography Mass Spectrometry (GCMS)

The structural elucidation and quantities of palmitic acid/palmitate (Hexadecanoic acid) extract of plantain stalk was investigated using Gas Chromatography Mass Spectrometry (GC-MS). The GC-MS results as presented in Table 1 shows the Retention time (RT) of various fatty acid methyl esters (FAMEs) present and also their relative abundance in respect to corrected area and peak heights respectively.

Table 1: Fatty acid composition of PSR

S/N	Compounds	Retention Time (mins)	%Area	Peak Height	%of total
1	Pentadecanoic acid, methyl ester	36.429	0.7	611762	0.781
2	Heneicosanoic acid, methyl ester	36.579	1.1	885033	1.140
3	Hexadecanoic acid, methyl ester	37.906	0.4	787408	0.450
4	9-Hexadecanoic acid, methyl ester	38.056	5.1	8245002	5.124
5	9-Octadecenoic acid, methyl ester	38.111	0.7	1337736	0.729
6	Hexadecanoic acid, methyl ester	38.283	13.0	22329292	13.036
7	Hexadecanoic acid, 14-methyl ester	38.872	0.9	759758	0.955
8	9-Octadecenoic acid, methyl ester	38.967	3.4	7392674	3.421
9	9,12-Octadecadienoic acid, methyl ester	39.524	`3.9	7621654	3.897
10	9-Octadecnoic acid, methyl ester	39.572	4.7	11281264	4.724
11	9-Octadecenoic acid, methyl ester	39.603	11.2	24666942	11.274
12	Heptadecanoic acid, 16-methyl-, methyl ester	39.729	2.6	5538678	2.685
13	Octadecanoic acid, 17-methyl-, methyl ester	40.106	0.5	1095507	0.594
14	Cyclopropanoeoctanoic acid, 2-Octyl-, methyl ester	40.208	1.1	2621950	1.096
15	Eicosanic acid, methyl ester	40.766	0.5	812386	0.573
16	Urs-12-en-24-oic acid, 3-oxo-, methyl ester	41.614	3.4	1566114	3.491

The GC-MS was able to elucidate about 31 fatty acids and 16 of them important to this research are presented in the Table 1. The successful extraction of palmitic acid/palmitate from plantain stalk was confirmed at line 6 with a RT of 38.283. However, hexadecanoic acid was also observed at line 3 while another isomer of the acid was also observed at line 7. To this effect, all the aforementioned line that affirmed palmitic acid showed a similarity index/minimum quality of 90% and above (≥90%).

% composition of hexadecanoic acid can be calculated thus:

$\%CH_3(CH_2)_{14}COOH$ = % of total (lines:6+3+7)

= 13.036+0.450+0.955

=14.4%

Therefore, the percentage of palmitic acid out of all compounds detected in the sample plantain stalk analyzed was calculated to be 14.4%.

Hence the total FAME present can be obtained as follows:

= lines 1+2+ -------- +16 (i.e. %of total lines 1-16)

=0.781+1.140+ ------ + 3.491

=53.97%

$$\frac{\%CH_3(CH_2)_{14}(COOH)}{\%\ of\ total\ FAME} = \frac{14.4415}{53.97}\ x\ \frac{100}{1} = 26.7\% \cong 27\%$$

It can be inferred from these results that the concentration of FAMEs in the extract is 27% out of which 14% is palmitic acid. This infers that half of the fatty acid content of of the extract belongs to palmitic acid.

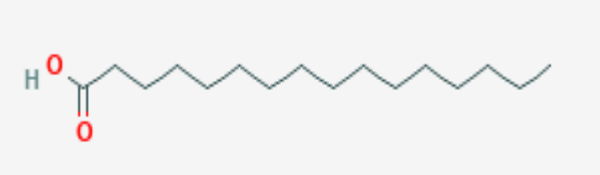

Figure 3: 2D Structure of Palmitic Acid

3.2 Fourier Transform Infra-Red Spectroscopy

An observation of the chemical structure of palmitic acid (Figure 3) reveals the presence of the following functional groups: C-C, C-H, C=O, C-O and O-H which can be observed by analyzing the extract sample with an

Infra-Red Spectrometer. The raw extract and the dissolved extracts were analyzed for their functional groups and the resulting spectra are shown in Figures 4-7.

The functional groups contained in palmitic acid and can be revealed under Infra-red radiation by absorptions or transmissions at wavelengths of 3300-3400cm^{-1} for OH stretch, 2900-2850cm^{-1} for C-H stretch, 27445-2710cm^{-1}for CHO (aldehyde) stretch, 1600-1700cm^{-1} for C=O stretch and 1750-1700cm^{-1} for C-C stretch. The absorption in the fingerprint region (1500-400cm^{-1}) of the spectra can be attributed to the functional group bending thereby confirming the functional groups observed in the stretching region. It can be observed from Figure 4 that the O-H and C-H stretches although small were observed at 3209cm^{-1} and 2937-2881cm^{-1} respectively for the raw plantain stalk extract. The small peak at 1610cm^{-1} may correspond to the presence of either C-C or C-O functional groups or both [15].

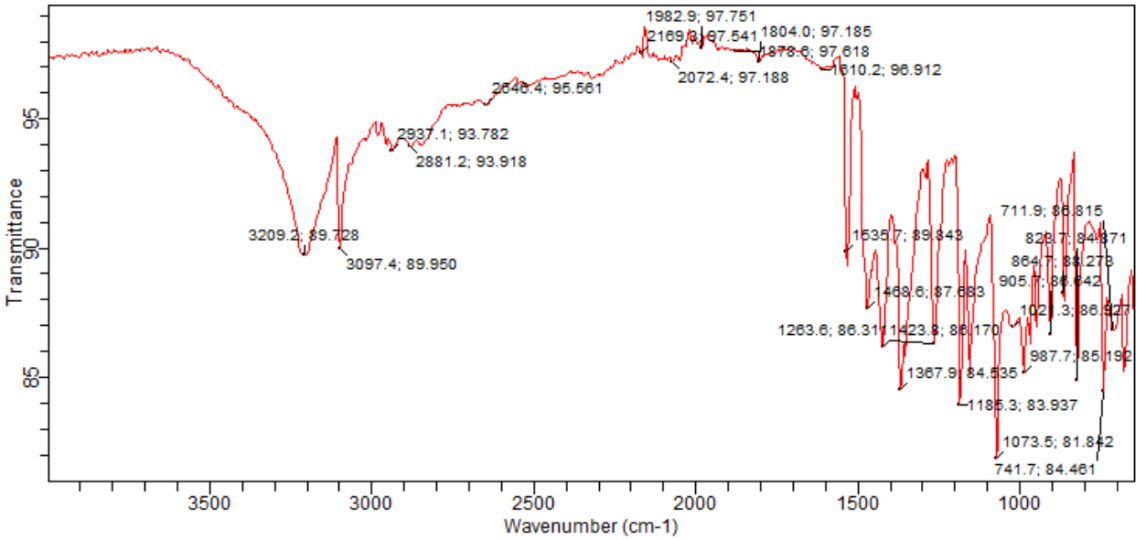

Figure 4: Spectra for Raw Extract

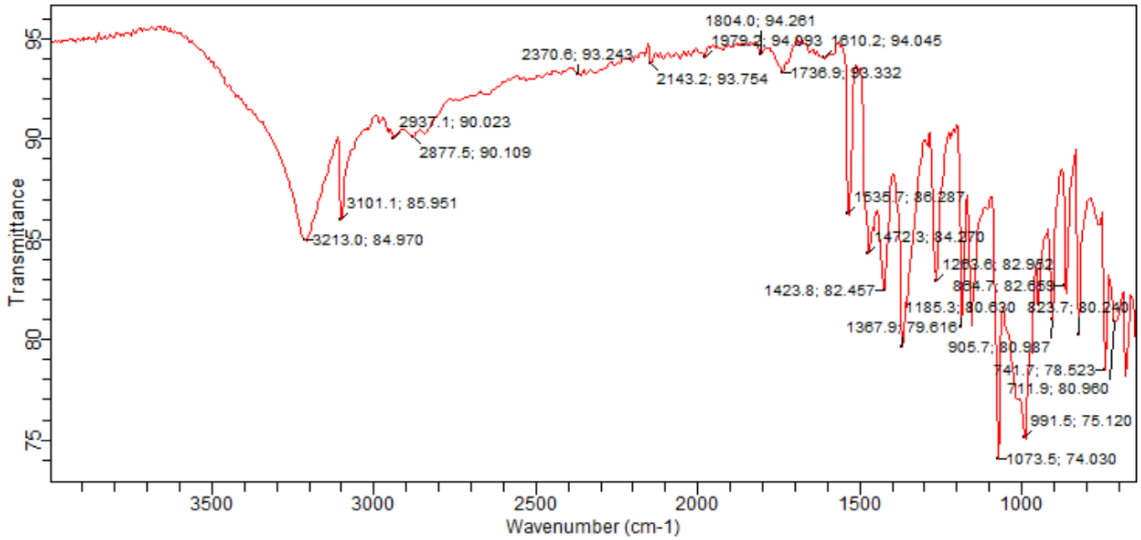

Figure 5: Spectra for Ethanol dissolved extract

The extract was dissolved in Hexane and Ethanol to increase the concentration of palmitic acid which is necessary for this study while removing some other unimportant components. The spectra of the ethanol dissolved extract as shown in Figure 5 is relatively the same for the untreated extract (Figure 4) with the exception of a small peak observed at 1736 cm^{-1} which could be attributed to the presence of C=0 stretch of a fatty acid [16]. The spectra of the hexane dissolved extract (Figure 6) shows a broader stretch at 2929cm^{-1} and at 1640cm^{-1} indicating the more pronounced presence of C-H and C=O stretches respectively.

These results infer that Hexane would selectively concentrate palmitic acid than ethanol or water. However, a closer look at Figure 7 would reveal that residue dissolution in acetone was more effective than that of hexane shows a much better result better result than hexane since the peaks indicating the presence of palmitic acid are stronger. As seen in Figure 5, the strong peaks at 2855.1cm-^{1} and 1744.4cm-^{1} can be attributed to the CH_2 symmetric stretching band and C=O stretching band of palmitic acid respectively [8].

It can then be inferred that Acetone would be a more suitable solvent to dissolve the plantain fiber residue in order to selectively concentrate palmitic acid.

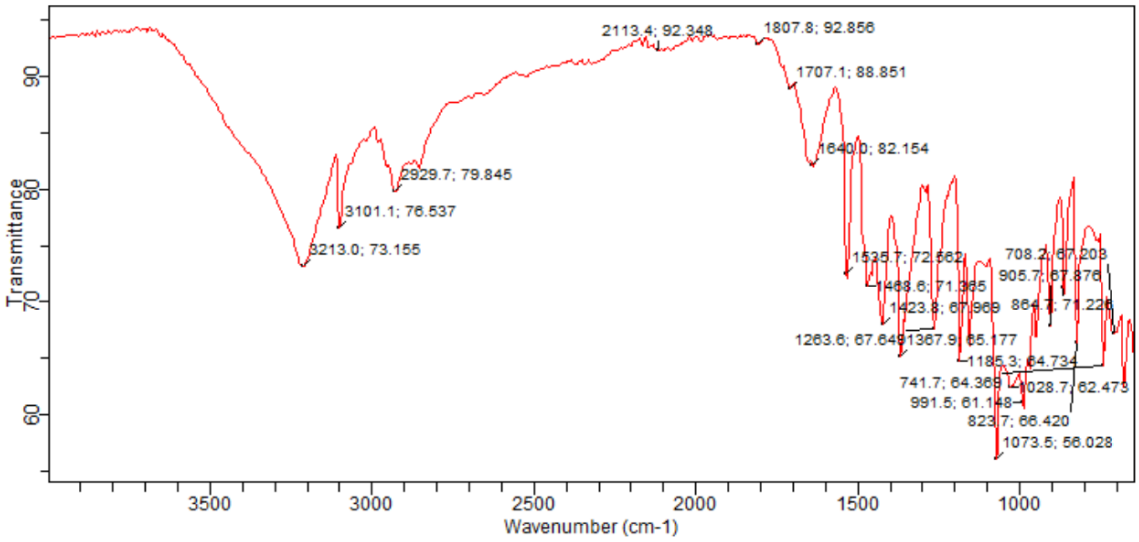

Figure 6: Spectra for Hexane dissolved Extract

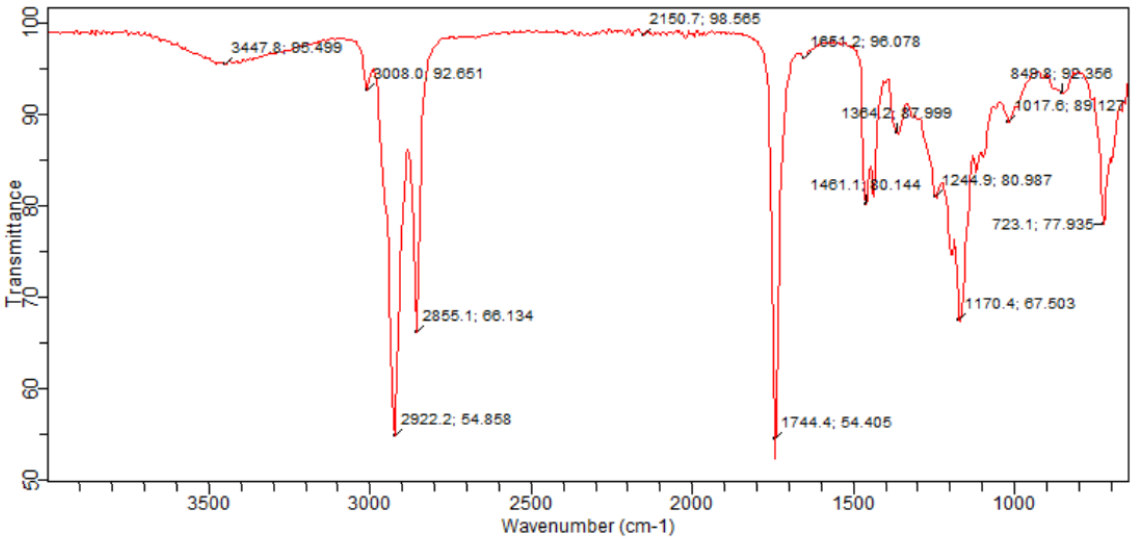

Figure 7: Spectra for Acetone-dissolved extract

3.3 X-Ray Fluorescence (XRF)

XRF is useful in the identification of inorganic components present in a material sample. The XRF analysis of the residue and dissolved extract samples identified about 24 elements of which 6 elements (Fe, K, Si, Cl, Ca and S) have higher compositions as compared to others. Figure 8 shows a graphical representation of the percentage composition of the 6 elements in the PSR and in the dissolved PSR extracts with NT denoting the originally extracted residue (PSR). Dissolution of PSR in acetone, hexane and water increases the composition of Si and Ca and reduces the composition of K present in the extract. Ethanol however reduced the Ca present in the extract to a level below detection. It can be observed from the figure that Si relatively has the highest composition in all the dissolved extracts. High composition of Ca in the residue makes it a potential material for liming [17].

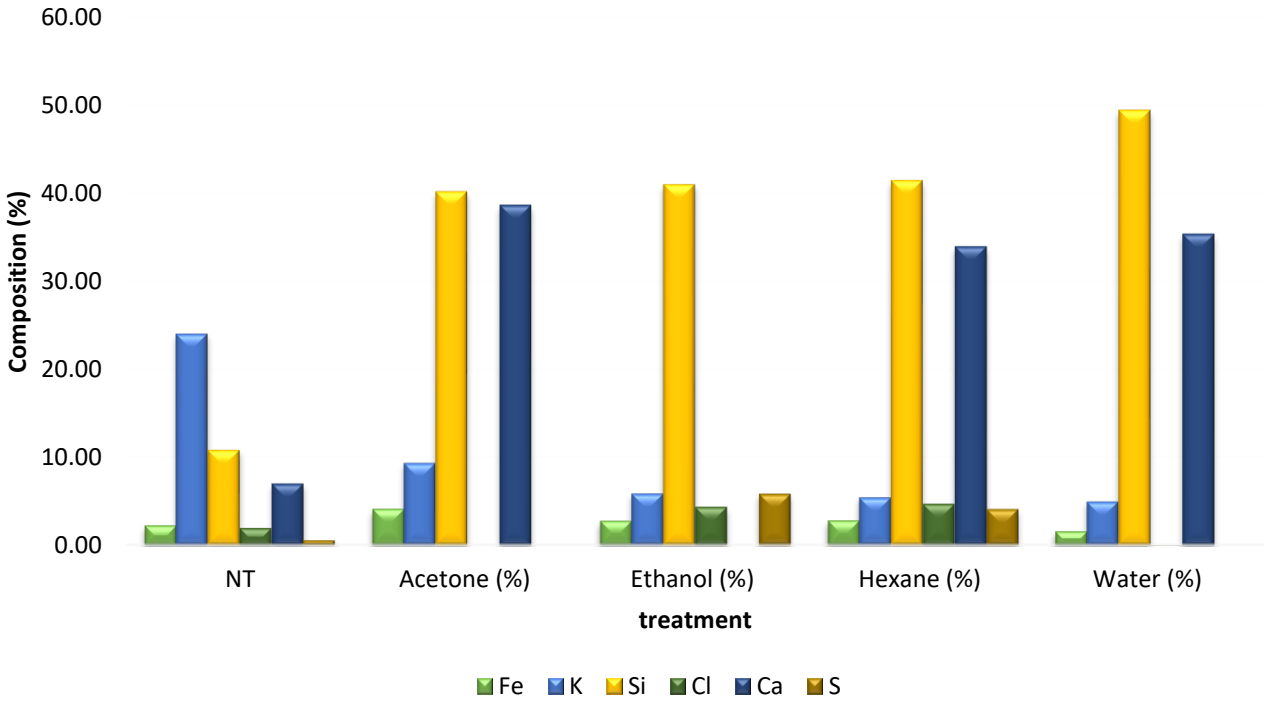

Figure 8: Elemental Composition

4. CONCLUSION

Residue obtained from Plantain stalk was analyzed for the presence of palmitic acid using GCMS spectroscopy and percentage composition of palmitic acid in the residue was found to be 14.4%. The raw extract was dissolved in four solvents: acetone, hexane, ethanol and water respectively and the dissolved extract after drying was analyzed for functional groups and elemental composition using FTIR spectroscopy and XRF respectively. FTIR results showed that acetone dissolved residue revealed a strong presence of the C=O and CH_2 bands of palmitic acid which implies a higher yield of palmitic acid compared to the extract dissolved in the other solvents. Thus acetone proved to be the most effective solvent in the extraction of palmitic acid from PSR.

NOTATIONS

PSR - Plantain Stalk Residue
NT - Non-Treated Residue
FAME - Fatty Acid Methyl Esters
Fe - Iron
K - Potassium
Si - Silicon
Cl - Chlorine
Ca - Calcium
S - Sulphur

REFRENCES

[1] Bulama, J., S. Dangoggo, M. Halilu, A. Tsaf, and S. Hassan, *Isolation and characterization of palmitic acid from ethyl acetate extract of root bark of Terminalia glaucescens.* Chem. Mater. Res, 2014. **6**: p. 140-143.
[2] Nwokonkwo, D. and G. Okeke, *The Chemical Constituents and Biological Activities of Stem Bark Extract of Theobroma Cacao.* Global Journal of Science Frontier Research: E Interdiciplinary, 2014. **14**.
[3] Calvo, B., I. Collado, and E.A. Cepeda, *Solubilities of palmitic acid in pure solvents and its mixtures.* Journal of Chemical & Engineering Data, 2008. **54**(1): p. 64-68.
[4] Khuwijitjaru, P., S. Adachi, and R. Matsuno, *Solubility of saturated fatty acids in water at elevated temperatures.* Bioscience, biotechnology, and biochemistry, 2002. **66**(8): p. 1723-1726.
[5] Kulkarni, S. and S. Fernando, *Separation of Palmitic and Palmitoleic Acid Using Transition Metal Salts: Experimental vs Theoritical Analyses.* International Research Journal of Pure and Applied Chemistry, 2015. **5**(1): p. 1.

[6] Gonçalves Bonassoli, A.B., G. Oliveira, F.H. Bordón Sosa, M.P. Rolemberg, M.A. Mota, R.C.a. Basso, L. Igarashi-Mafra, and M.R. Mafra, *Solubility Measurement of Lauric, Palmitic, and Stearic Acids in Ethanol, n-Propanol, and 2-Propanol Using Differential Scanning Calorimetry*. Journal of Chemical & Engineering Data, 2019.
[7] Sbihi, H.M., I.A. Nehdi, S. Mokbli, M. Romdhani-Younes, and S.I. Al-Resayes, *Hexane and ethanol extracted seed oils and leaf essential compositions from two castor plant (Ricinus communis L.) varieties.* Industrial Crops and Products, 2018. **122**: p. 174-181.
[8] Iwai, Y., M. Uno, H. Nagano, and Y. Arai, *Measurement of solubilities of palmitic acid in supercritical carbon dioxide and entrainer effect of water by FTIR spectroscopy.* The Journal of supercritical fluids, 2004. **28**(2-3): p. 193-200.
[9] Park, S., F. Viklund, K. Hult, and R.J. Kazlauskas, *Vacuum-driven lipase-catalysed direct condensation of l-ascorbic acid and fatty acids in ionic liquids: synthesis of a natural surface active antioxidant.* Green Chemistry, 2003. **5**(6): p. 715-719.
[10] Garlapati, C. and G. Madras, *Solubilities of palmitic and stearic fatty acids in supercritical carbon dioxide.* The Journal of Chemical Thermodynamics, 2010. **42**(2): p. 193-197.
[11] Rosales-García, T., J.M. Rosete-Barreto, A. Pimentel-Rodas, G. Davila-Ortiz, and L.A. Galicia-Luna, *Solubility of Squalene and Fatty Acids in Carbon Dioxide at Supercritical Conditions: Binary and Ternary Systems.* Journal of Chemical & Engineering Data, 2017. **63**(1): p. 69-76.
[12] Tenbohlen, S. and M. Koch, *Aging performance and moisture solubility of vegetable oils for power transformers.* IEEE transactions on power delivery, 2010. **25**(2): p. 825-830.
[13] Cadena, E.M., J.M. Vélez, J.F. Santa, and V. Otálvaro, *Natural fibers from plantain pseudostem (Musa Paradisiaca) for use in fiber-reinforced composites.* Journal of Natural Fibers, 2017. **14**(5): p. 678-690.
[14] Imoisili, P.E., O. Fadare, A. Popoola, and A. Okoronkwo, *Effect of Chemical Treatment on the Morphology and Mechanical Properties of Plantain (Musa paradisiaca) Fiber.* 2017.
[15] Bunaciu, A.A., H.Y. Aboul-Enein, and Ş. Fleschin, *Vibrational spectroscopy in clinical analysis.* Applied Spectroscopy Reviews, 2015. **50**(2): p. 176-191.
[16] Tong, D.G., D.M. Tang, W. Chu, G.F. Gu, and P. Wu, *Monodisperse Ni 3 Fe single-crystalline nanospheres as a highly efficient catalyst for the complete conversion of hydrous hydrazine to hydrogen at room temperature.* Journal of Materials Chemistry A, 2013. **1**(21): p. 6425-6432.
[17] Zolue, G.M., *Characterization of biochar prepared from three different feed stocks*. 2013, University of Ghana.