

# Characterization of Native and Modified Fermented Smoked Cassava for Food Packaging Application

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## Abstract

The goal of this study is to see what changes may be made to fermented smoked cassava (FSC) for use in food packaging. For 5 days, cassava was soaked in water. The fermented cassava was shaped into balls and dried at 50°C in an oven. The fermented smoked cassava that resulted was milled. The fermented smoked cassava was subjected to chemical modification by pregelatinized phthalation. To make pregelatinized phthalated, FSC, the FSC was pregelatinized by heating the solution above 70°C and esterified with phthalic anhydride. The characteristics of modified and native FSC were investigated. The native FSC typical spherical form had vanished, and a fibrous-like irregular structure had replaced it, according to the results of scanning electron microscopy. After some alterations The degree of crystallinity in the native FSC has changed to amorphosity in the modified FSC, according to X-Ray Diffractometry. Thermogravimetry findings demonstrated that the native structural matrix of FSC was totally collapsed at 380°C, whereas the modified FSC structural matrix fully collapsed, leaving just residues at 700 °C. The modified fermented, smoked-dried cassava was discovered to be a promising alternative biopolymer for food packaging applications.

**Keywords:** Crystallinity; Modification; Phthalic anhydride; Pupuru; Fermented smoked cassava.

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## 1. Introduction

Cassava (*Manihot esculentus*) is one of the most stable food crops in tropical regions [1] and is an essential food crop because of its stress tolerance, efficient food energy production, and availability ideal for current farming and food systems in Africa [2]. It is a root starchy crop that is a major source of food security in Africa due to its disease and drought resilience, as well as its capacity to grow in poor soil [3]. Cassava tubers are highly perishable and cannot be kept in good condition for an extended period of time after harvesting without deteriorating in quality. Cassava is processed into dried products in various ways to fit the taste, local needs, and storage requirements in order to extend its shelf life. Cassava tuber processing is divided into four categories: meal, flour, chips, and starch [4]. It's made into a variety of meals, including fufu, garri, lafun, and Fermented Smoked Cassava (pupuru). It is ground into flour, which is then used to make a variety of dishes or consumed as a boiled, food gel, fried, or pounded food product [5].

Pupuru is a cassava flour that has been fermented and is similar to fufu. It comes from the Ijaje people of Nigeria's Ondo State's riverine region. Cassava flour (pupuru) is a fermented cassava meal consumed by between 4 and 6 million Nigerians [6]. It is, also known as "Ikwurikwu," is a common staple meal for those living in the riverine portions of Nigeria's southern, western, eastern, and middle belts [7]. Cassava processing to Fermented Smoked Cassava is a substantial source of income for certain Nigerians and plays a vital part in ensuring food security [8] The processing procedure distinguishes Fermented Smoked Cassava from other cassava fermented goods such as fufu, gari, akpu, and lafun [9].

The activities of lactic acid bacteria on the carbohydrate content of the cassava tuber have been blamed for acid generation during cassava fermentation [10]. Lasekan, *et al.* [11], found that adding soya beans to Fermented Smoked Cassava (FSC) increased its nutritional value. Chemical composition of cassava product was altered by processing method, according to Ayankunbi, *et al.* [12]. According to Osundahunsi and Oluwatoyin [13], drying processes for Fermented Smoked Cassava reduced the hydrogen cyanide content to a safe level. Modification increased the pasting and functional qualities of pupuru, according to Adewumi, *et al.* [14].

Fermented Smoked Cassava (FSC) is mostly consumed as a meal. Despite the fact that fermented Smoked Cassava has always been used as a food, it can also be a reliable replacement for petroleum-based polymer in a wide range of applications in the food packaging business, reducing the environmental difficulties that petroleum-based

polymer causes. Because of its reduced functional characteristics, native FSC may be underutilized [14, 15]. As a result, modification is critical to improve functional features, create new products, and expand applications, particularly in the food packaging industry [16]. Physical, chemical, or enzymatic modification of starch is required to change the physicochemical properties of unmodified (native) starch in order to obtain superior derivatives that have advantages over their native forms [17]. This research focuses on the effects of modifying fermented, smoked-dried (FSDC) cassava for use in food packaging.

## 2. Materials and Methods

### 2.1. Materials

In the year 2020, tubers of cassava (*Manihot esculentus*) were purchased at the local farm in Ado –Ekiti, Ekiti State, Nigeria. All of the reagents were purchased from Sigma Aldrich and were of analytical quality.

### 2.2. Methods

#### 2.2.1. Fermented Smoked Cassava Processing

The traditional process for producing "pupuru," as described by (Shittu *et al* ) [18]. A farm in Ado-Ekiti provided fresh cassava roots that were 11-12 months old. Fresh cassava roots (*Manihot esculenta*) were peeled, rinsed, and chopped into little 8cm long pieces before steeping in water for 5 days. The fermented cassava was mashed by hand, then dewatered, formed into balls, and dried in a 50°C oven. Drying was carried out until a consistent weight was achieved. The fermented smoked-dried cassava (FSC) balls that resulted were scraped, pulverized, and packaged in various materials.

#### 2.2.2. Preparation of Pregelatinized Phthalated FSC.

The Silvia, *et al.* [19] approach was used. Following the process of pregelatinized starch phthalate was obtained in two phases (gelatinization and esterification) as follows: 100 g FSC was dispersed in 500 ml distilled water for the gelatinization procedure. The solution was heated above 70°C before being dried into flakes in a 50°C oven, milled, and sieved. The Fermented Smoked Cassava pregelatinizes were then disseminated in distilled water before being used. The esterification reaction was carried out by dispersing 10g of pregelatinized FSC in 100ml of distilled water with 16.7% phthalic anhydride solution in ethanol 96%. The pH of the reaction was kept constant at 8-10 by adding 10M NaOH solution on a regular basis. Following the addition of phthalic anhydride, the reaction was allowed to run for 30 minutes with continuous stirring, and the solution was allowed to stand overnight. The pH of the solution was then adjusted to 6.5–7.0 by adding 1M HCl solution. After drying, milling, and sieving the precipitate, pregelatinized phthalated FSC was obtained.

#### 2.2.3. Scanning Electron Microscopy (SEM) with Energy Dispersive X-ray Spectroscopy (EDX)

A scanning electron microscope was used to analyze the morphology of the starch granules. A scanning electron microscope (Hitachi SU8030 FE-SEM Tokyo, Japan) was used to examine the form and size of the native and modified FSC granules at an accelerating potential of 5.0 kV. Prior to inspection, all samples were sputter-coated with Au/Pd.

#### 2.2.4. X-Ray Diffraction

An X-ray diffractometer was used to record the X-ray diffraction pattern using a copper anode x-ray tube (Cu-K1 radiation) (Rigaku D-max Tokyo, Japan). The starch powders were securely packed in sample holders, and each sample was subjected to a 40 kV, 20 mA X-ray beam. At a step size count of 2 s, the scanning region of the diffraction angle ( $2\theta$ ) was from 5° to 60°.

#### 2.2.5. Thermal Analysis

The thermal analysis was carried out with a Shimadzu TG-DTA model TG-60 thermogravimetric analyser (Shimadzu, Kyoto, Japan). The tests were carried out in a synthetic air atmosphere with a flow rate of 100 mL/min and a heating rate of 10°C/min in opened alumina crucibles containing roughly five milligrams of starch. The experiments began at 30 degrees Celsius and ended at 600 degrees Celsius.

## 3. Results and Discussion

### 3.1. Scanning Electron Microscopy (SEM) with Energy Dispersive X-ray Spectroscopy (EDX)

Scanning Electron Microscopy (SEM) was used to examine the surface morphology of native and modified FSC. As illustrated in Figure 1a, the natural FSC granules have a consistent spherical form and are strongly linked together. In Fig. 2b, you may see a micrograph of a phthalated FSC. The regular spherical form of the native FSC vanished after phthalation, and a fibrous-like irregular structure replaced the globular structure of the native FSC. Bean starches are oval to spherical in shape and come in a variety of sizes, according to Granza, *et al.* [20]. The intermolecular hydrogen bonds that were broken during the phthalation process were responsible for the changes in

granular structure of phthalated FSC. After acetylation, Yixiang, *et al.* [21] found that the starch granule structure of maize was lost.

### 3.2. Energy Dispersive X-Ray Spectroscopy (EDX)

The elemental composition of the native and modified Fermented Smoked Cassava is shown in Figures 2a and 2b. Carbon (C) and oxygen (O) were found in the elemental composition of the native fermented smoked cassava (Fig. 2a). Hydrogen, on the other hand, does not appear on the spectrum because it lacks a core electron that the machine can detect. The elements C, O, Na, and Cl were found in the EDS of the phthalated Fermented Smoked Cassava (Fig. 2b). Because NaOH and HCl were employed to modify the pH during modification, Na and Cl appear in the spectrum.

Adewumi, *et al.* [14], reported the FTIR spectra of native and phthalated pupuru. Peaks at 3425.58 cm<sup>-1</sup> and 2926.07 cm<sup>-1</sup> belong to O-H and C-H stretching, respectively, whereas peaks at 1654.92 cm<sup>-1</sup> and 1438.18 cm<sup>-1</sup> relate to O-H and C-H bending, respectively. Due to the carbonyl group of the ester, phthalated Fermented Smoked Cassava revealed additional absorption bands at 1932.67 cm<sup>-1</sup> (evidence that phthalation reaction had occurred).

### 3.3. X-Ray Diffraction

The XRD tests were taken to see if chemical treatment had any effect on the crystallinity of the native pupuru. Figures 3a and 3b show the XRD patterns of native and phthalated pupuru. The native Fermented Smoked Cassava (3a) XRD pattern exhibited significant peaks at 17.5°, 20°, 21.5°, and 27.5° (2θ). The phthalated FSC XRD pattern (Figure 3b) revealed a collapse of crystallinity with a faint peak at 23° (2θ). The phthalated amorphous form was caused by the collapsing crystallinity. X-ray diffraction demonstrates that throughout the esterification process, the native pupuru's crystalline structures were disrupted, and a new FSC structure was produced. It's possible that the phthalation took place in the FSC crystalline area. According to Luo and Shi [22] the partial replacement of hydroxyl groups with acetyl groups during acetylation results in disordering of the crystalline structure, which lowers inter and intramolecular hydrogen bonding.

### 3.3. Thermogravimetric Analysis

Figures 4a and 4b show the thermographic analysis results of the native and phthalated pupuru. At temperatures below 100°C, native Fermented Smoked Cassava (Figure 4a) began to lose weight, indicating that water content in the granules was being removed. At 280°C, the second weight loss was seen, owing to the degradation of organic materials in the pupuru. The structural matrix has totally crumbled at 380°C, leaving just the remnants. The initial weight loss for phthalated Fermented Smoked Cassava (Figure 4b) started while the temperature was below 100°C, indicating that the granules were losing water. At 280°C, the second weight loss occurred, owing to the degradation of organic materials in the starch derivative. At a temperature of roughly 320°C, the weight of the Phthalated Fermented Smoked Cassava was significantly reduced in the third stage. The structural matrix has fully collapsed at 700°C, leaving the remnants. Because there are fewer free hydroxyl groups available after phthalation. The phthalated Fermented Smoked Cassava is more thermally stable than the original pupuru. Phthalate substitution on starch resulted in phthalated Fermented Smoked Cassava with a greater molecular weight, requiring a higher temperature to breakdown. As a result, phthalation improved the thermal stability of the starch product.

## 4. Conclusion

Phthalic anhydride was used to successfully modify Pupuru. The phthalation process changed the shape of the granules. Crystallinity was collapsed due to alteration, according to XRD data. Thermodynamic analysis revealed that phthalated Fermented Smoked Cassava had improved thermal stability. The qualities of Fermented Smoked Cassava were improved as a result of the changes. This approach offers an appealing alternative in order to broaden its industry applications.

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Figure-1a. Scanning electron micrograph of native FSC

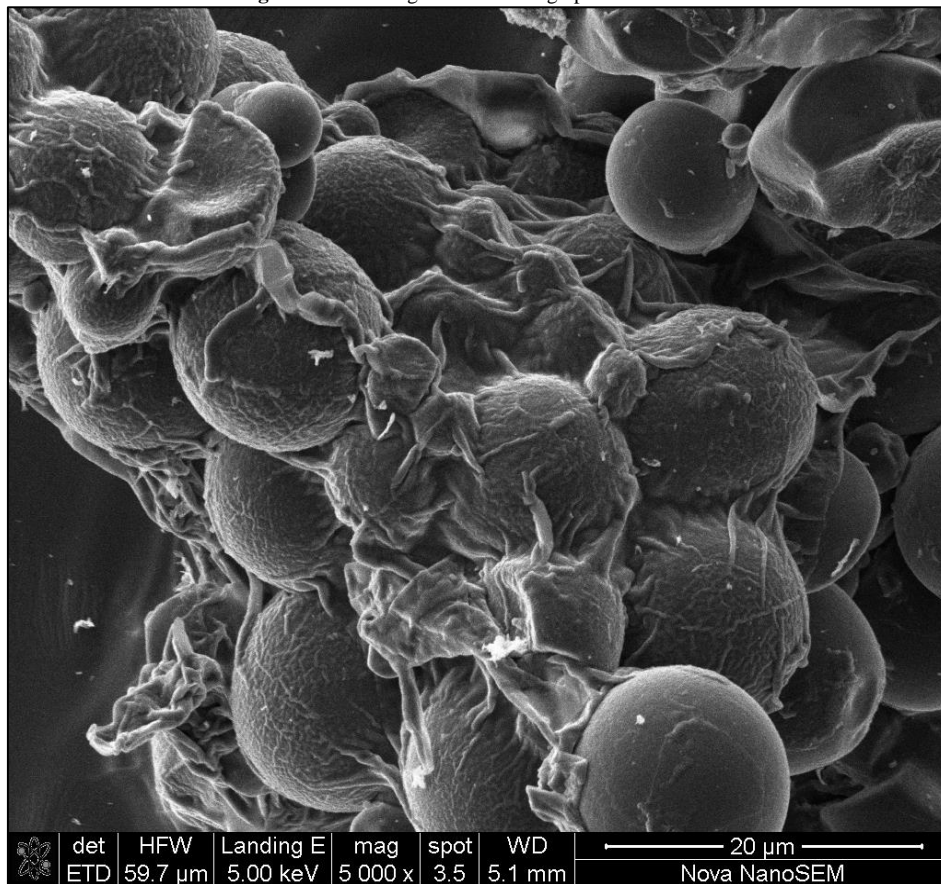


Figure-1b. Scanning electron micrograph of phthalated FSC

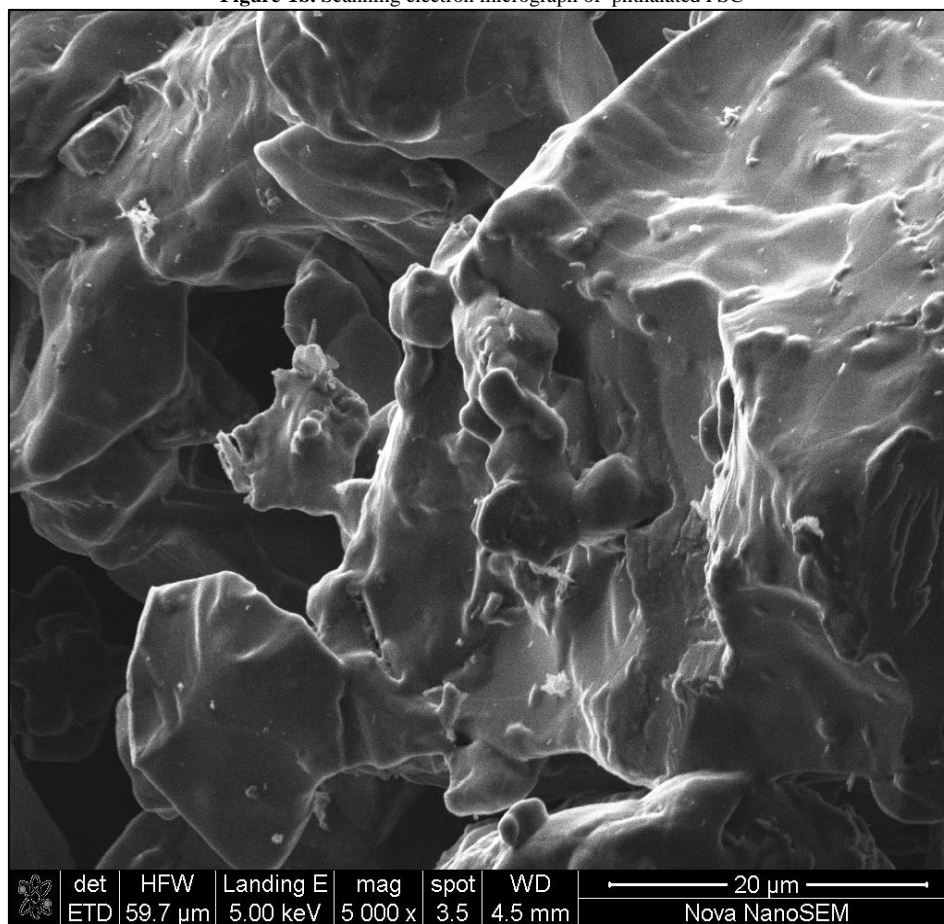


Figure-2a. Energy Dispersive X-ray of native FSC

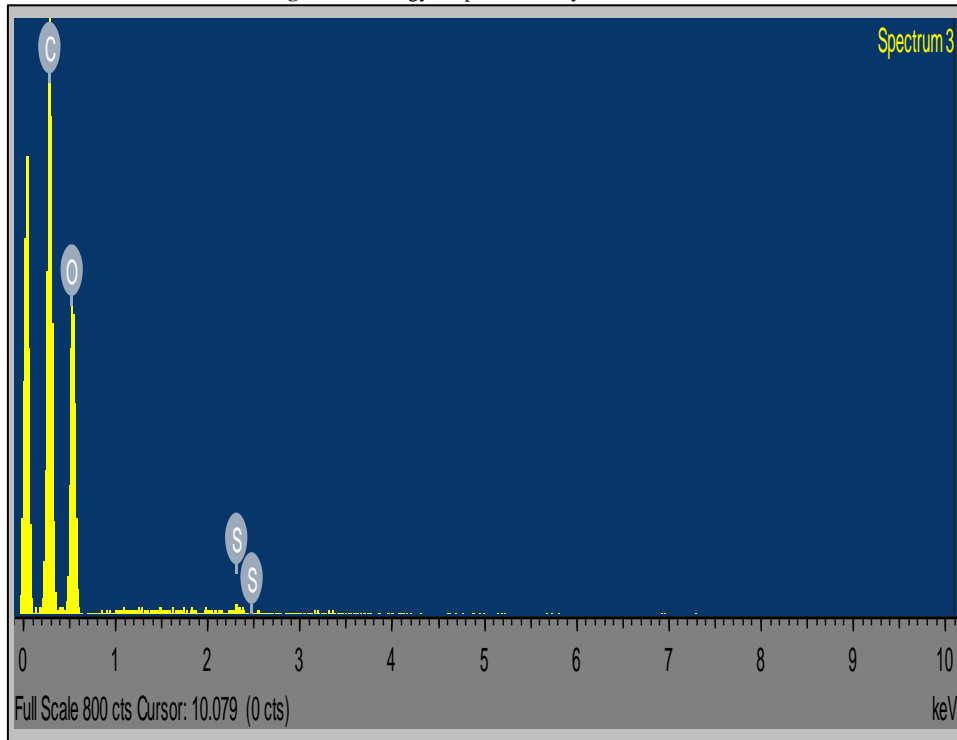


Figure-2b. Energy Dispersive X-ray of phthalated FSC

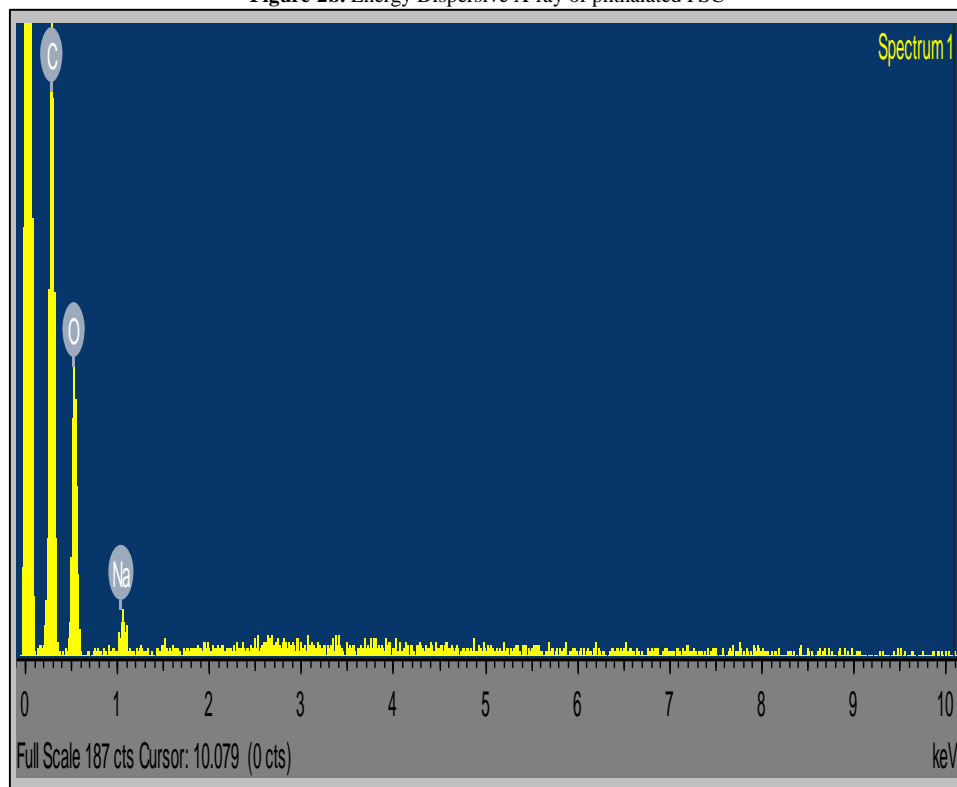


Figure-3a. X-ray diffraction spectrum of native FSC

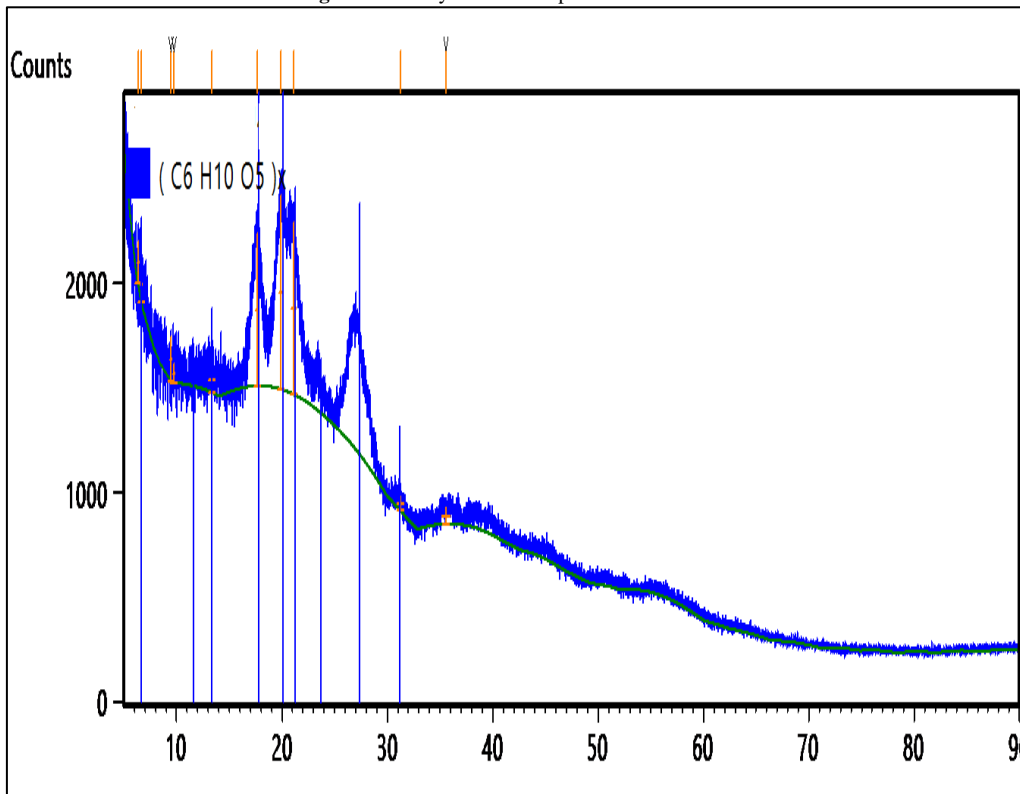


Figure-3b. X-ray diffraction spectrum of phthalated FSC

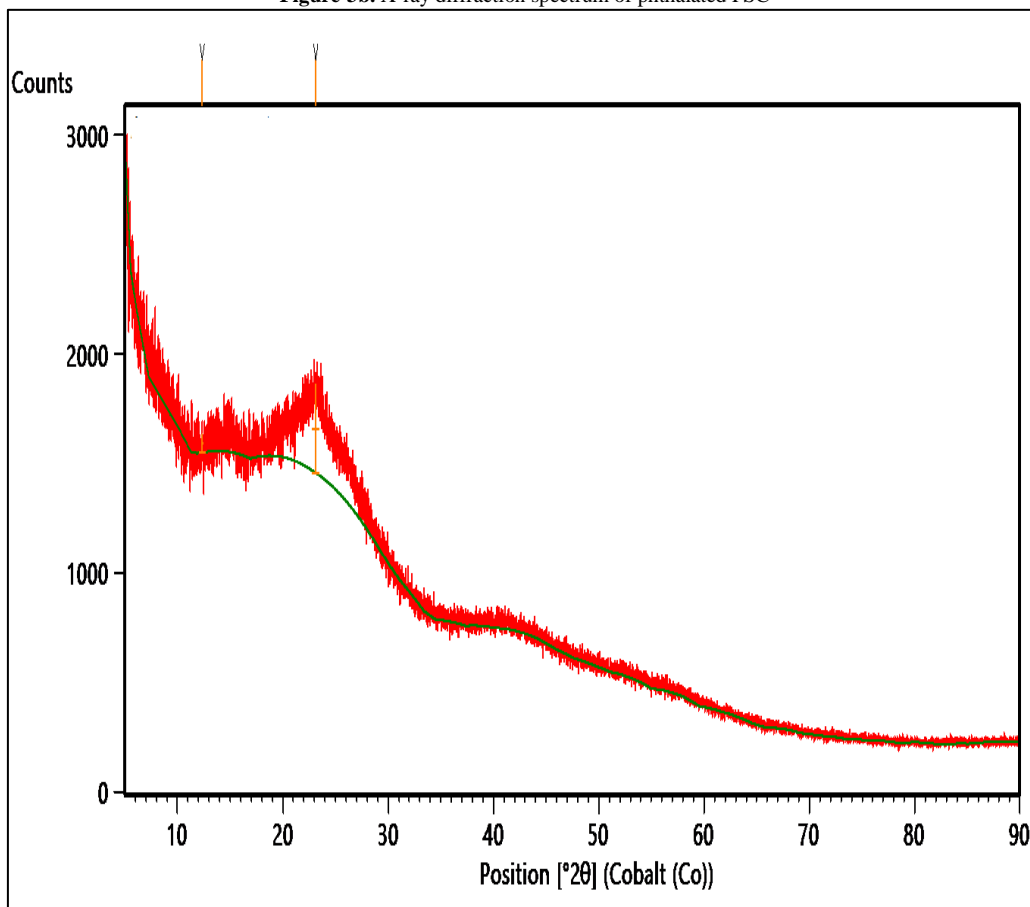


Figure-4a. Thermogravimetry analysis (TGA) of native FSC

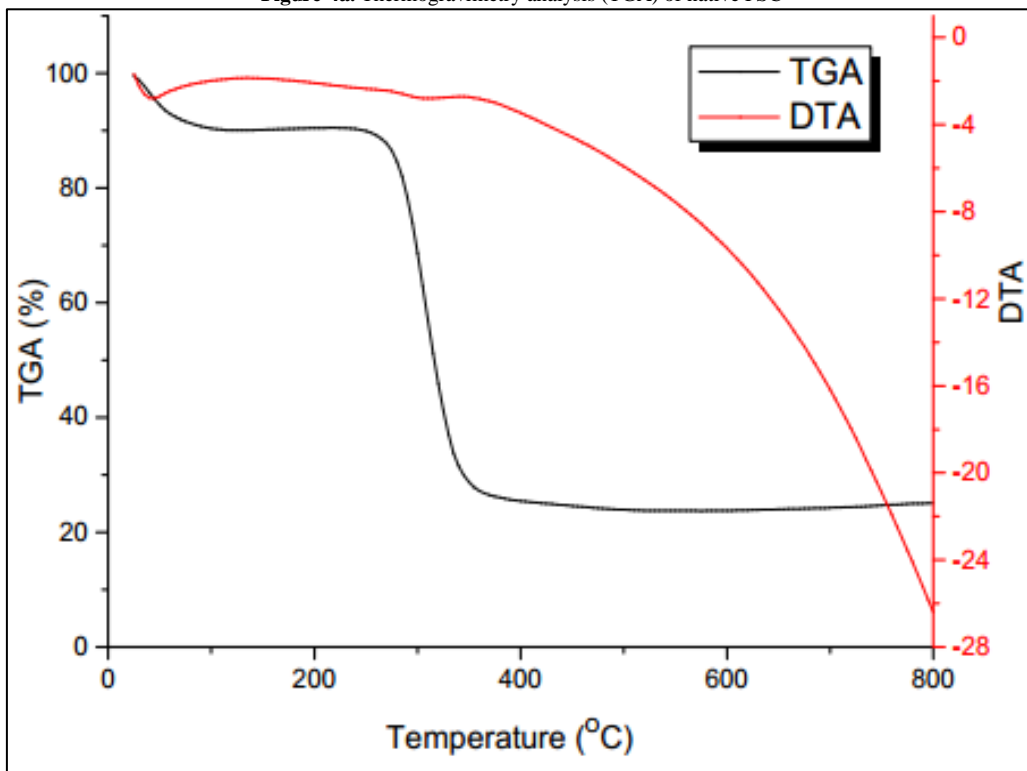


Figure-4b. Thermogravimetry analysis (TGA) of phthalated FSC

