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Extraction and Characterization of Starch from Pineapple Stem: A Sustainable Approach

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ABSTRACT

Pineapple stems are currently a substantial material for agriculture all over worldwide because pineapple needs to be replaced on an annual basis. This organic waste can be a good source of starch. The aim of the research is to extract and evaluate starch using traditional extraction technique. The pineapple stem starch was characterized using Fourier transform infrared spectroscopy (FTIR), Scanning electron microscopy (SEM), Energy dispersive X-Ray analysis (EDX), X-Ray diffraction (XRD) and Thermogravimetric analysis (TGA). Iodine test confirms the presence of starch. The concentration of starch present in the pineapple stem was determined by anthrone method. FTIR confirmed that the peak obtained represent the characteristic feature of polysaccharide. The scanning electron microscopy study reveals that the stem starch is round and polyhedral in shape. The EDX analysis shows that the pineapple stem starch was made up of carbon and oxygen. According to XRD studies the stem starch has B type crystal in amorphous phase. Thermogravimetric study reveals that the degradation started at lower temperature. When compared to commercial starches, pineapple stem starch has unique characteristics. Pineapple stem starch is an excellent source of resistant and flexible starch for culinary and other products usage due to these prospective favourable qualities.

Key words: Pineapple stem, Starch, Extraction, Characterization, FTIR, SEM, XRD, TGA analysis

1. Introduction

The pineapple (*Ananas cosmosus* L.) belongs to the family *Bromeliaceae*, considered as one of the most widely consumed tropical fruits in the world. With over 2.7 million tons of pineapple generated, the Philippines leads all other exporters (Kumar *et al*., 2023 and Dhar *et al*., 2023). Costa Rice, meanwhile comes in second with over two million tons. India is one among the largest producers of pineapple and they produce various processed goods, such as jam, jelly, juice, marmalade, etc (Purkait *et al*., 2023). After the harvesting, huge quantities of pineapple stems are discarded as agricultural waste (Sarangi *et al*., 2023). Every two years, pineapple plants need to be replanted (Hernosa *et al*., 2002). According to estimates, one pineapple plant produces 6 to 8 kilogram of debris after harvest, comprising leaves, stems, and roots (Apipatpapha *et al.,* 2022). Despite the fact that pineapple leaves are suggested as a fibrous reinforcement of composite polymers, 0.6 kilograms of pineapple stems are formed and typically thrown away after harvesting (Eixenberger *et al*., 2022).

Farmers commonly disposes pineapple stem waste by flaming and sun-drying which harms the atmosphere. To tackle these issues, investigations of pineapple waste have been conducted by various researchers in order to recycle it value added products (Tangsrianugul *et al*., 2023). In order to transform pineapple waste into a variety of industrially significant compounds and products, a circular bioeconomy method based on the integration of several process technologies are necessary, which will lead to enhanced economic growth and a waste free environment (Nath *et al*., 2023). In the past decade, research into the development of green materials has increased in order to lower the impact of human activity on the environment (Bradu *et al*., 2022).

The agricultural waste of pineapple stem exhibits significant potential in the starch replacement. After cellulose, starch is the most prevalent biomolecule on the planet and the primary carbohydrate reserve in plants (Aili Hamzah *et al*., 2021). Pineapples are well-known for their high levels of vitamin C, fibre and a digestive enzyme called bromelain (Sarangi *et al*., 2023). Furthermore, pineapple stems are rich in fibre, used as a raw material for extracting starch, bromelain enzyme and bioactive compounds (Nath *et al*., 2023). The usage of chemically modified starches has decreased, hence the demand for natural starches has increased globally (Surendren *et al*., 2022). There are numerous uses for native starches in the food, pharmaceutical, paper, cosmetic and other industries (Pirsa and Hafezi., 2023).

Waste generated from pineapple fruit, through using appropriate extraction procedure to obtain significant biological and nutritional compounds a major challenge in the context of sustainable waste management. Various pineapple waste products, including the stem, core, skin, crown etc., contain considerable levels of high-value bioactive chemicals. The sustainable isolation of starch from pineapple stem waste is a great technique to reduce agricultural waste whilst obtaining potential benefits from nature (Kumar *et al*., 2024).

Starches and other biomaterials have been essential in delivery of therapies throughout the last 30 years (Hasanin., 2022). The majority of stretched snacks are made from starch-rich raw materials (rice, wheat, maize, cassava, potatoes, and oats), particularly wheat or corn flour, due to their excellent capacity for expansion and widely favoured product texture. For the manufacturing of unique non-gluten- extruded snacks, pineapple stem starch can be employed as a potential ingredient in place of commercial starches (Dias Capriles *et al*., 2023).

Starch from pineapple stem extracted conventionally and purified simply without the use of chemicals or laboratory processes (Huang *et al*., 2022). On dry basis pineapple stem consist of 97.8% indicating the high purity of starch with highest gelatinization temperature and increased concentration of amylose. These distinctive and special properties indicate the possible use of pineapple stem starch with thermoplastic properties for food and industrial application (David Troncoso *et al*., 2022). The dry weight extraction yield of pineapple stem starch was 30%. Pineapple stem starch granules had a semi-angular shape with partially circular portions that had multiple smooth surfaces with a median particle size of 9.69 mm (Sriprablom *et al*., 2023).

Thus, this research aimed to obtain starch from pineapple stem and characterize the extracted stem starch using scanning electron microscopy (SEM), X-ray diffraction (XRD), Fourier transform infrared (FTIR) spectroscopy and TGA analysis that would provide information about its potential application in dietary supplementation for humans.

2. Methodology

2.1 Extraction of starch from pineapple stem

Pineapple stem was obtained from farm after harvesting pineapple fruit in Kanyakumari, Tamil Nadu. To remove soil and other debris it was washed thoroughly with water and peel of the skin. Surface sterilised with sodium hypochlorite solution and washed thrice with distilled water. The stem was cut into small pieces and grinded with distilled water using mixer grinder. Filtered using muslin cloth and it was repeated for several times until the slurry’s milkiness was eliminated or diminished, then the slurry was centrifuged at 10000 rpm for 10 minutes and the fluid was retained. The pellet was purified with alcohol followed by distilled water and dried at hot air oven for 24 hrs. The powder obtained was sieved and stored in desiccators.

2.2 Characterization of starch

2.2.1 Iodine test:

Iodine test is performed to determine the presence of starch. 1g of starch and 15 mL of water was heated and cooled. To 1 mL of sample add 0.1N 0.5 mL Iodine solution and the change in colour were observed (Halick and Keneaster., 1956).

2.2.2 Estimation of starch by anthrone method

The concentration of starch present in the pineapple stem was done by anthrone method using glucose as standard and the absorbance was measured at 630 nm (Viles and Silverman., 1949).

2.2.3 Fourier transform infrared spectroscopic analysis (FTIR)

The functional group of the extracted pineapple stem was investigated using FTIR spectroscopy. The FTIR analysis method scans the test materials and examines chemical characteristics using infrared light. 1-2 mg of powdered materials was analysed in the frequency range from 4000 to 400 cm^{-1} with the resolution of $4cm^{-1}$.

2.4 SEM and EDS analysis

The surface morphology of the stem starch was characterized using scanning electron microscopy (MIRA3 TESCAN), using a secondary electron detector with 10.0 kV in Bharat Ratna Prof. CNR Rao Research Centre. The element present in the extracted pineapple stem starch was determined using EDS analyser in the resolution of 127.4 eV.

2.5 X-ray diffraction (XRD)

X-ray diffractometer was used to evaluate the crystallinity of stem starch (XPERT-PRO). A scanning speed of 10 deg/min was used with a radiation angle of 2θ adjusted between 5^0 and 60^0C.

2.7 Thermogravimetric analysis

Thermogravimetric analysis was performed using TGA EXSTAR 6300 with 5 mg of starch in an aluminium pan at a scanning speed of 10 ° C per min ranging from 25 ° C to 300 ° C.

Derivative thermogravimetric curves were plotted in order to determine the inflection points, or the temperature during which the most reduction in weight occurs.

3. Result and Discussion

3.1 Extraction of starch from pineapple stem

The pineapple stem with an average weight of 500 g that yields 55g of starch per plant (Rinju and Harikumaran., 2021). This is significant when you consider the huge amount of pineapple agricultural waste that accumulate annually (Fig 1). Pineapple stem has 9% of starch on wet basis. The starch was extracted from pineapple stem without using any chemicals. Rinju and Harikumaran reported that we can generate at least three tons of starch per acre of pineapple farm if we create an affordable and environment friendly method for extracting starch from agriculture waste.

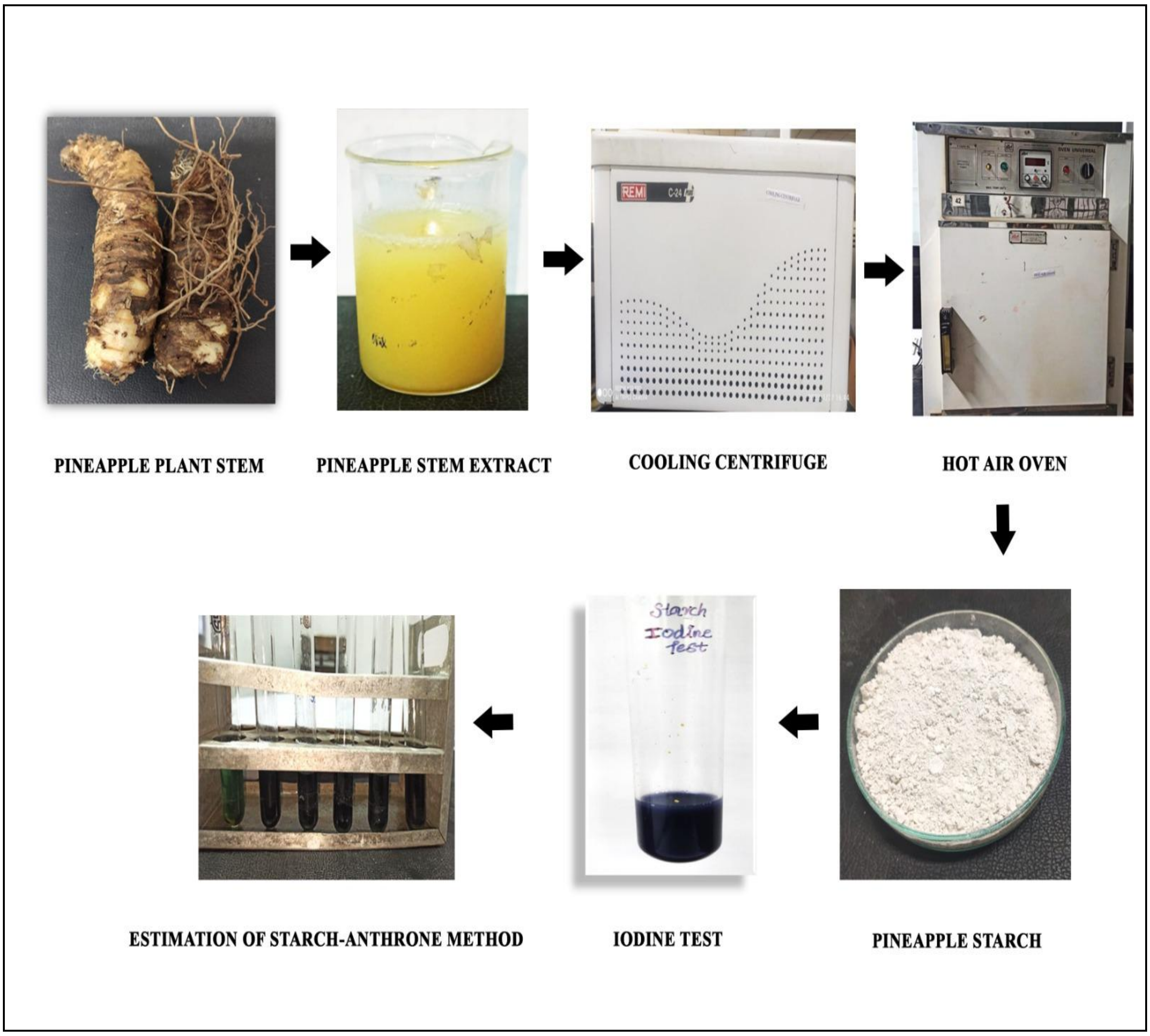

Figure 1: Extraction of starch from pineapple stem

3.2 CHARACTERIZATION OF STARCH

3.2.1 Iodine test

Iodine test confirms the presence of starch. The pineapple stem starch with iodine turns into deep blue colour, which indicates the presence of starch (Fig 1). The amylose in starch forms helices where iodine molecules can bound, forming a dark blue or black colour (Halick and Keneaster., 1956).

3.2.2 Estimation of starch by anthrone method

Glucose molecule reacts with anthrone reagent to give a blue- greenish colour in acidic conditions (Fig 1). The concentration of starch in the pineapple stem was determined to be 11.5 % ± 0.72 mg/mL.

3.2.3 Fourier transform infrared spectroscopic analysis (FTIR)

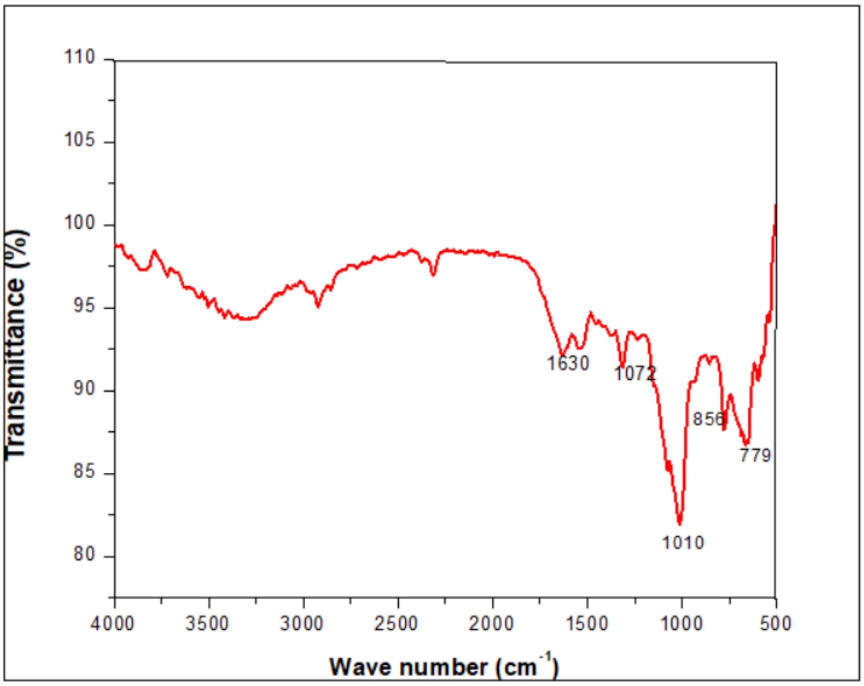

Figure 2: FTIR spectroscopy of pineapple stem starch

The peak obtained at 1630 cm^{-1} is due to the C=C stretching of alkene and the peak at 1072 cm^{-1} is responsible for C-O stretching of carbohydrate in the stem starch. The other peaks around the 1010 cm^{-1} peak considered to be distinctive feature of polysaccharides 856 cm^{-1} is bending of -C=O inorganic carbonate and CH stretching of 779 $cm^{-1}$is due to amide group in the pineapple stem starch (Fig 2). Radi and Abedi reported that all starches exhibit C-O-H stretching at 1100-900 cm^{-1}.

3.2.4 SEM and EDS analysis of pineapple stem starch

The morphology of stem starch was tiny round granules having flat surface without any pores that were polyhedral shape with acute angles and edges (Fig 3). The findings of the scanning electron microscopy investigation shows that the distribution of granule shape is not uniform. The starch in tubers and roots contain granules of this type. This result is in agreement with Rinju and Harikumaran 2021. The existence of starch constituents was confirmed by energy dispersive X-ray analysis. The peak around 5.76 keV and 5.12 keV correlates to the C and O binding energies (Fig 4).

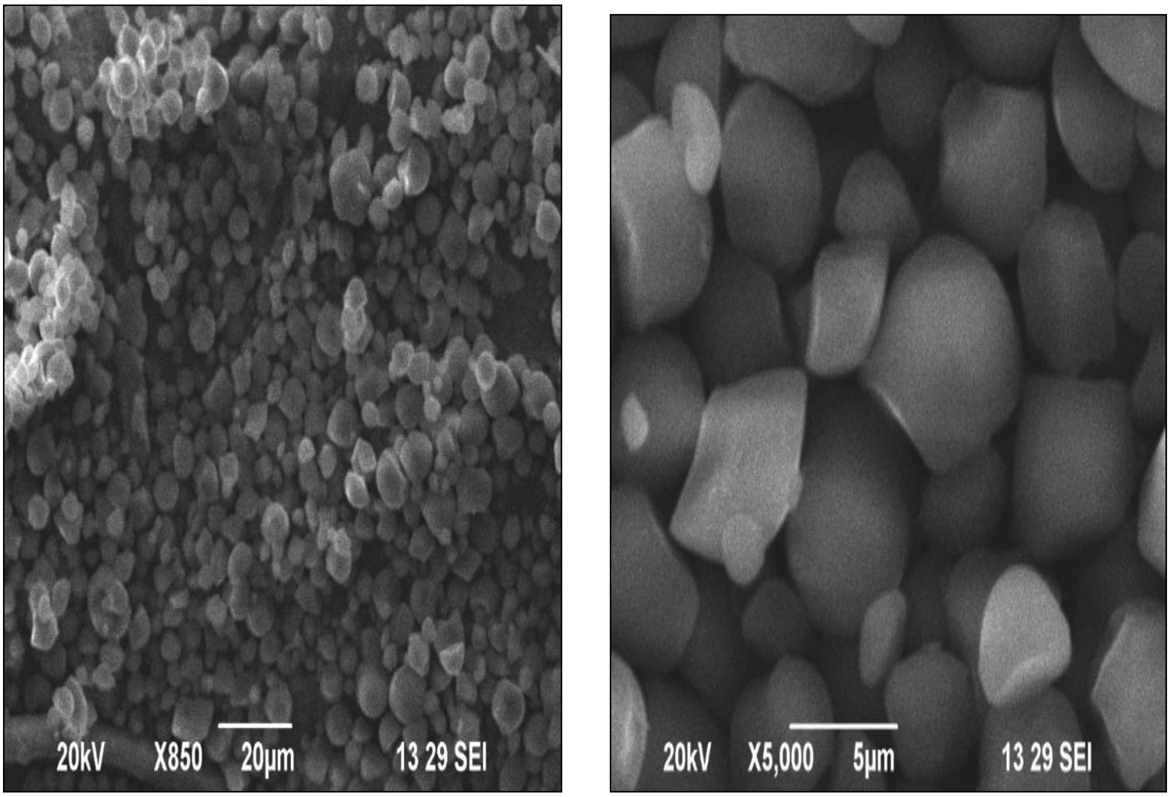

Figure 3: SEM analysis of pineapple stem starch

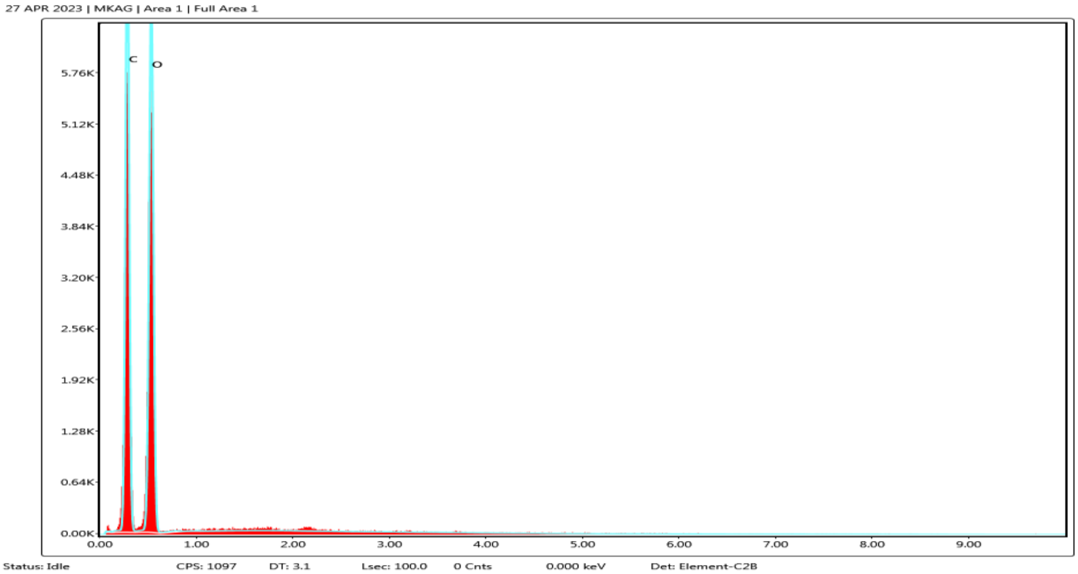

Figure 4: EDS of pineapple stem starch

3.2.5 X-ray diffraction (XRD) analysis of pineapple stem starch

The XRD analysis of stem starch reveals that it possesses B type crystal in amorphous phase, which is particularly found in tuber and root starch. A type starch was found mainly in cereals where C type is the mixture of A & B type starch (Fig 5). Tarique reported that since pineapple stem starch originates from the stem, it was predicted that it would follow the "B type" diffraction pattern. The amorphous phase is linked to the short branching chains of amylopectin, the ordered phase is mostly determined by the amylose and amylopectin branching chains (Malik *et al*., 2023).

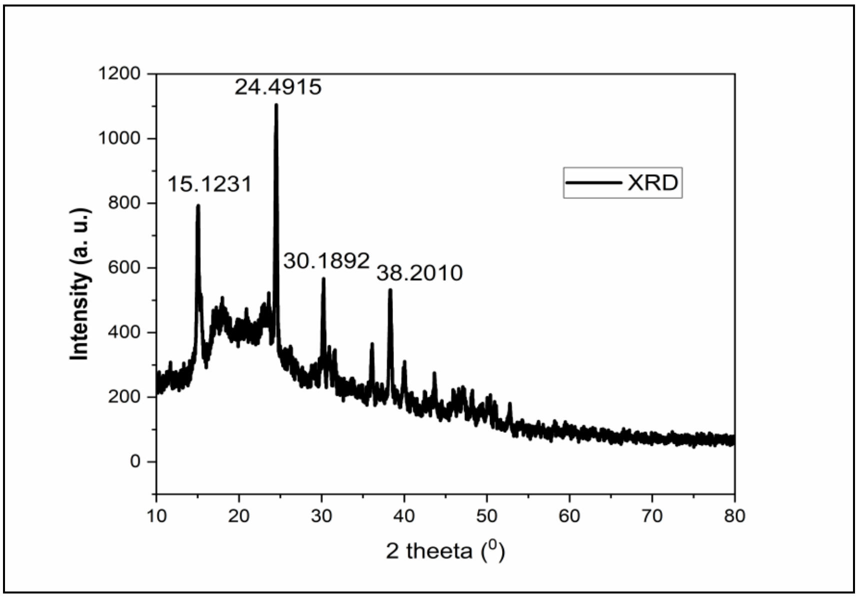

Figure 5: XRD of pineapple stem starch

3.2.6 Thermogravimetric analysis

TGA analysis reveals that the degradation occurs at lower temperature. During this stage, the mass loss of the pineapple residual 4.42% respectively (Fig 6). The highest degradation starts at stage II which release the majority of volatile substance around 50% of total loss. For pineapple residue, the major temperature was 150-360^{o} C. Due to the removal of a very small amount of water by evaporation and dehydroxylation processes, the initial weight loss was noticed at a temperature below 100^{o} C (Nurazzi et al., 2021).

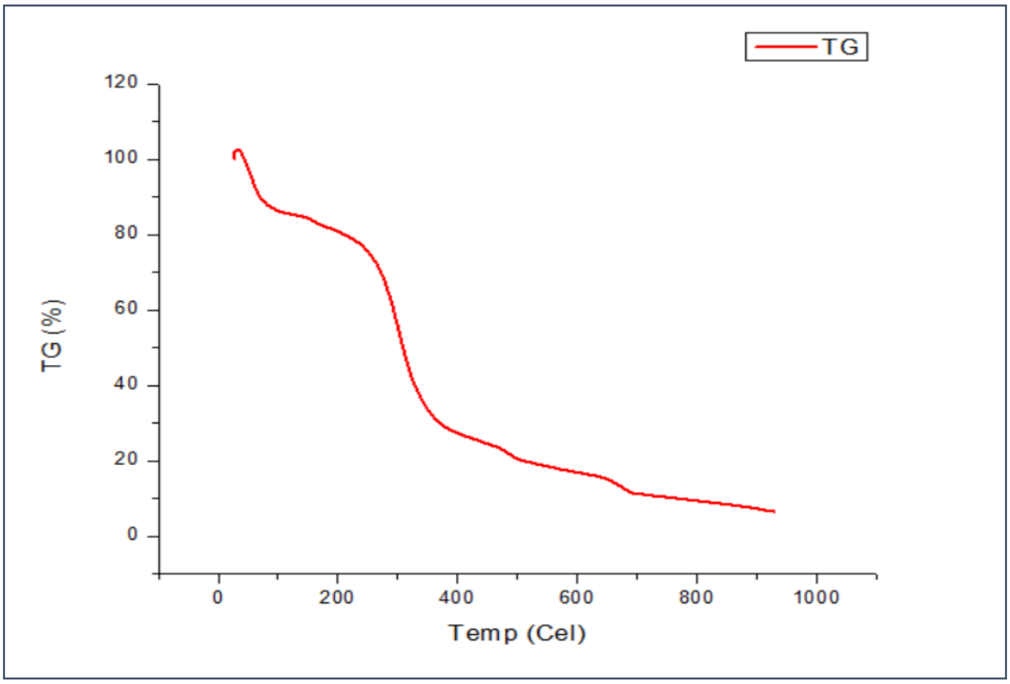

Figure 6: TGA of pineapple stem starch

4. Conclusion

Pineapple has been a valuable fruit over the years due to its distinct aroma, numerous volatile chemicals, and nutritional value. This research highlights the use of trash to make food and other products as a means of promoting innovative and sustainable development. Pineapple waste contains many reusable chemicals with excellent economic value. Waste generated from pineapple fruit, through using appropriate extraction procedure to obtain significant biological and nutritional compounds a major challenge in the context of sustainable waste management. Various pineapple waste products, including the stem, core, skin, crown etc., contain considerable levels of high-value bioactive chemicals. Utilizing this agricultural waste starch contributes in giving farmers an increase in income. We truly anticipate that it will benefit society and boost the nation's and farmers' economic prosperity.

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