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Modification of Elephant Foot Yam Starch by Pregelatinization Treatment and to Study the Physiochemical Properties of Starch

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ABSTRACT:

The present study aims to explore the extraction, purification, thermal modification, and characterization of starch obtained from the fruit tuber of Amorphophallus paeoniifolius (Elephant Foot Yam), an underutilized yet promising starch source. Native starches often exhibit limitations such as poor solubility, low thermal stability, and inadequate flow properties, which restrict their broader application in pharmaceutical, food, and industrial sectors. To overcome these challenges, a pregelatinization treatment was employed at controlled temperatures (62°C, 65°C, and 68°C) to enhance the physicochemical and functional properties of the isolated starch.

The extraction was carried out using wet milling followed by Soxhlet purification. The modified starches were evaluated for solubility across different solvents, bulk and tapped densities, angle of repose, Hausner's ratio, and Carr's index to assess powder flow characteristics. Structural transformations were further analyzed using Fourier Transform Infrared Spectroscopy (FTIR) and X-ray Diffraction (XRD). The results revealed that pregelatinization significantly enhanced the solubility, flow properties, and crystallinity profile of the starch. Notably, starch modified at 68°C demonstrated the most favorable characteristics, including highest bulk density (0.8233 g/cm³), lowest Carr's index (10.001%), and complete transition from a semi-crystalline to an amorphous structure.

This study confirms the potential of Elephant Foot Yam starch as an alternative biopolymer with improved functional attributes suitable for diverse applications. The findings also emphasize the effectiveness of physical modification through pregelatinization as an eco-friendly, cost-efficient method for upgrading native starch properties without chemical interventions.

KEYWORDS: Elephant Foot Yam (Amorphophallus paeoniifolius), Pregelatinization, Modified Starch, Physicochemical Properties, FTIR, XRD, Powder Flow Properties, Starch Characterization, Thermal Modification, Alternative Biopolymer

1. INTRODUCTION

Starch is regarded as one of the most significant products derived from plants for human consumption. This polysaccharide serves as a primary energy reservoir for individuals globally and

is synthesized as a storage carbohydrate within various plant species. The process of photosynthesis yields approximately 2,850 million tons of starch on an annual basis. The predominant sources of starch utilized by humans include cereals, while roots and tubers also hold substantial importance. The annual production of starch from cereal crops is estimated to be around 2,050 million tons, with roots and tubers contributing approximately 679 million tons (Burrell, M.M. et al., 2003). Starch is a ubiquitous carbohydrate, ranking just behind cellulose and chitin in terms of natural prevalence (Bertoft, E. 2017). As the principal storage carbohydrate in numerous plants, starch not only serves as a crucial energy source for developing seeds but also possesses significant value in both food and non-food industries due to its distinctive structural properties. (Stasiak, M**.** et al., 2013). It stands out as one of the most essential yet adaptable food ingredients, characterized by qualities that facilitate a myriad of industrial applications. Commercial sources of starch for industrial use include cereal grains such as corn, wheat, and sorghum, as well as tubers and roots like potatoes, tapioca, and arrowroot. Cereal grains, pseudo-cereals, legumes, roots, and tubers represent the primary raw materials for starch extraction. Starch plays an integral role in determining the textural properties (including thickness, creaminess, or firmness) and sensory characteristics (such as mouthfeel, consistency, and visual appeal) of processed food products. For instance, it functions as a thickening agent in sauces, contributing to their smooth and creamy texture (Tharanathan, R. N. et al., 2005). The application of starch in the food industry is extensive, serving to thicken, gel, stabilize, and prolong the shelf life of food items. Consumers favor food starches due to their accessibility, affordability, and unique functional attributes. Natural starch is a versatile ingredient utilized in the production of modified starches, sweeteners, and ethanol. From a chemical perspective, starch is composed of two glucan polymers, namely amylose and amylopectin. These polymers are organized within granules of varying sizes designated as large A, medium B, and small C types as well as in distinct shapes, including disk-like and spherical forms. The starch granules are structured in semi-crystalline and amorphous concentric layers. Starches derived from various sources, as well as individual types of starch, exhibit variations in their chemical composition (including alpha-glucans, moisture levels, lipids, proteins, and phosphorylated residues) and the structural characteristics of their components, which are correlated with the surface properties, hardness, and crystallinity of the starch granules. The physical and chemical attributes of starch granules are fundamental in determining their behavior and functionality. A comprehensive understanding of these properties is essential for selecting the appropriate starch for specific applications and for making informed decisions regarding modifications needed to achieve the desired characteristics for particular uses (Cornejo-Ramírez et al., 2018). The inherent versatility of starch is frequently constrained by its intrinsic properties, necessitating modifications to enhance its functionality in order to fulfill consumer demands.

Native starch, which includes issues like poor solubility, high swelling power, and inadequate functional properties. These limitations can hinder its effectiveness in food technology, necessitating modifications to enhance its performance. The starch modification focuses on various physical modification method, including Heat Moisture Treatment (HMT),
annealing, and gelatinization. These methods aim to improve physico-chemical, functional, structural and thermal properties of native starch. The modifications are essential for overcoming the inherent limitations of starch, thereby increasing its applicability in food products. The results indicates that all physical modifications positively influenced the functional and physiological characteristics of

native starch. Notably the HMT resulted in most significant restrictions in swelling power, solubility, moisture content, and water absorption capacity (Gunaratne, 2023).

1.1 COMPOSITION OF STARCH:

Starch is a complex carbohydrate (polysaccharide) composed of glucose units linked together. It serves as the primary energy storage molecule in plants. The structure of starch consists of two main components: amylose and amylopectin.

A. Amylose:

Amylose is a linear or slightly branched polymer of α-D-glucose units. The glucose molecules are connected by α-(1→4) glycosidic bonds. Due to its linear structure, amylose can form helical structures, making it less soluble in water. Typically, amylose constitutes 20-30% of starch.

B. Amylopectin:

Amylopectin is a highly branched polymer of α-D-glucose units. It has α-(1→4) glycosidic bonds in the linear chains and α-(1→6) glycosidic bonds at the branch points (every 24-30 glucose units). The branched structure makes amylopectin more soluble in water and provides rapid enzymatic breakdown. It makes up 70- 80% of starch.

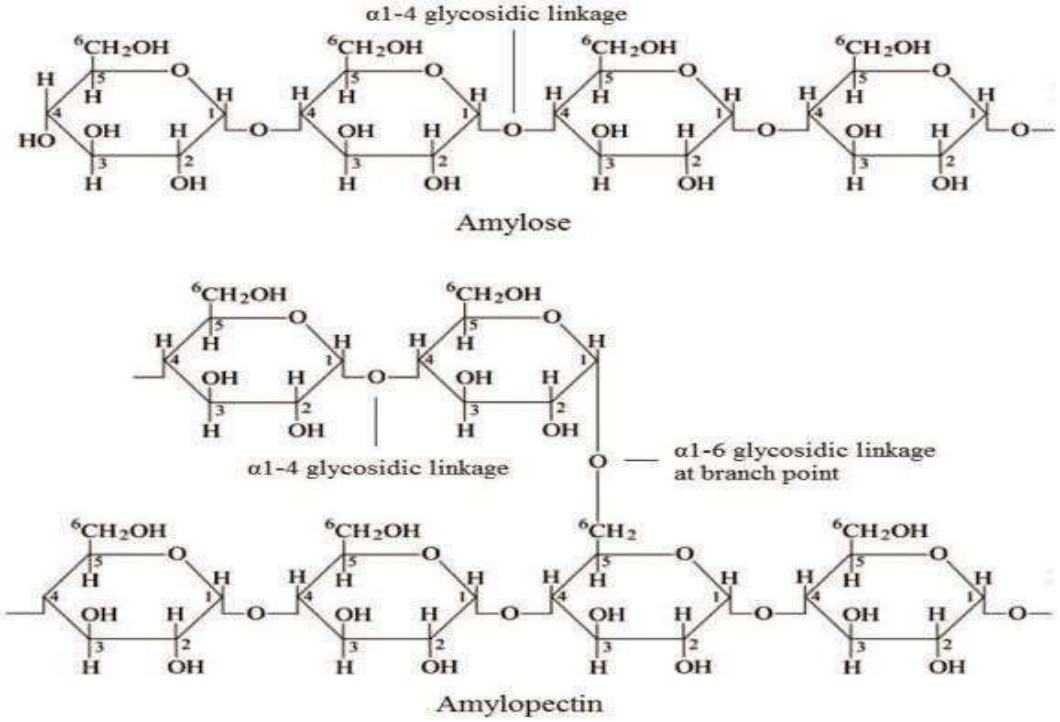

Fig.1.1: Structure of Amylose and Amylopectin.

1.2 GRANULAR STRUCTURE:

Starch is stored in plants as granules, with varying sizes and shapes depending on the plant source (e.g., rice, corn, potato). These granules exhibit a semi-crystalline structure, contributing to their resistance to digestion.

The crystalline structure of starch granules can be classified into three major types based on X-ray diffraction patterns: A-type, B-type, and C-type starch structures. These structural types differ in their molecular arrangement, water content, and sources.

1. **A-Type Starch:**

Found in cereal starches (e.g., wheat, rice, maize). Has a tightly packed crystalline structure with a low water content. Forms a more compact and ordered structure due to the close packing of amylopectin side chains. Exhibits stronger interactions between starch molecules, making it more resistant to enzymatic degradation. X-ray diffraction pattern shows strong reflections at 15°, 17°, 18°, and 23° (2θ angle).

2. **B-Type Starch:**

Found in tubers and high-amylose starches (e.g., potato, banana). Has a more open crystalline structure with a higher water content. Contains longer amylopectin branch chains compared to A-type starch, leading to a looser packing. More susceptible to enzymatic hydrolysis than A-type starch. X-ray diffraction pattern shows characteristic peaks at 5.6°, 17°, 22°, and 24° (2θ angle).

3. **C-Type Starch:**

Found in legumes and some root starches (e.g., peas, beans, tapioca) Considered a hybrid structure, containing both A-type and B-type crystallinity. Exhibits intermediate properties in terms of water content and enzymatic digestibility. X-ray diffraction pattern shows a combination of A-type and B-type peaks Cornejo-Ramírez et al., 2018 and Tester, R. F. et al., 2004.

1.3 CLASSIFICATION OF STARCH:

Starch can be classified based on various factors such as its botanical source, structural properties, and functional characteristics. Below is a detailed classification:

I. Based on Botanical Source:

1. **Cereal Starch:** Derived from grains such as wheat, rice, maize, and barley. Typically contains a higher proportion of A-type crystalline structure.
   Example: Corn starch, Wheat starch.
2. **Root and Tuber Starch:** Derived from underground storage organs such as potato, tapioca, and yam. Typically has a B- type crystalline structure with higher water content.
   Example: Potato starch, Cassava starch.
3. **Legume Starch:** Derived from beans, peas, and other legumes. Exhibits a C-type crystalline structure, which is a combination of A-type and B-type structures. Example: Pea starch, Bean starch.
4. **Fruit Starch:** Found in fruits like banana and plantain. Often shows B-type crystalline characteristics.

II. Based on Molecular Structure:

1. **Amylose-Rich Starch:** Contains >30% amylose. More resistant to swelling and digestion; used for making resistant starch Example: High-amylose corn starch.
2. **Amylopectin-Rich Starch (Waxy Starch):** Contains >95% amylopectin. Has improved water absorption, swelling capacity, and thickening properties.

Example: Waxy maize starch.

III. Based on Crystalline Structure:

1. **A-Type Starch:**

Found in cereals like wheat, maize, and rice. Has a tightly packed structure with low water content. More resistant to enzymatic digestion.

2. **B-Type Starch:**

Found in tubers like potato and banana. Has a loosely packed structure with higher water content. Has a loosely packed structure with higher water content. Easily gelatinizes and swells.

3. C-Type Starch:

Found in legumes like peas and beans. Exhibits characteristics of both A-type and B-type starch.

IV. Based on Functional Properties

1. Native Starch:

Unmodified, naturally occurring starch. Used in food, pharmaceutical, and industrial applications.

2. Modified Starch:

Chemically, physically, or enzymatically treated to enhance desired properties. Examples include pregelatinized starch, acetylated starch, and cross-linked starch.

3. Resistant Starch:

Resistant to digestion in the small intestine, acting like dietary fiber. Beneficial for gut health and blood sugar control.

V. Based on Granule Size:

1. Large Granule Starch (10–100 µm):

Example: Potato starch, Wheat starch.

2. Medium Granule Starch (5–10 µm):

Example: Corn starch, Tapioca starch.

3. Small Granule Starch (1–5 µm):

Example: Rice starch, Amaranth starch.

VI. Based on Industrial Applications:

1. **Food Starch** – Used in thickening, stabilizing, and texturizing food products.
2. **Pharmaceutical Starch** – Used as a binder, disintegrant, or filler in tablets.
3. **Industrial Starch** – Used in textiles, adhesives, paper coatings, etc.

2. PLANT PROFILE

2.1 ELEPHANT FOOT YAM FRUIT:

Elephant Foot Yam (Amorphophallus paeoniifolius) is a tuberous plant widely used in traditional medicine and as a food source. Its pharmacognostic profile includes botanical description, macroscopic and microscopic characteristics, physicochemical parameters, and phytochemical constituents Kumar, V. et al., 2012. While its tuber is rich in starch and widely used, the starch content of its fruit has been less explored Patil et al., 2020. Starch extracted from the fruit of Elephant Foot Yam has potential applications in food, pharmaceuticals, and industrial sectors due to its unique physicochemical properties Reddy et al., 2014. The fruit of Amorphophallus paeoniifolius is not commonly consumed but contains a significant amount of starch. It is widely cultivated in Asia, Africa, and Pacific Island. Elephant Foot Yam is highly nutrious, monocotyledonous robust herbaceous tuber crop grown in tropical and subtropical region (Behera et al., 2016).

Fig 2.1: Elephant Foot Yam Fruit

2.2 Synonyms of Elephant Foot Yam (Amorphophallus paeoniifolius):

Scientific Synonyms: Amorphophallus campanulatus Blume ex Decne, Amorphophallus rivieri var. campanulatus (Blume ex Decne.) Engl**.**

Common Names in Different Languages: English: Elephant Foot Yam, Whitespot Giant Arum **Hindi:** Suran, Jimikand

Sanskrit: Surana, Suvarna, Kandalu

Tamil: Senaikizhangu **Telugu:** Kanda, Peddakanda **Malayalam:** Chena

Kannada: Suvarna gedde

Bengali: Ol **Marathi:** Suran **Gujarati**: Suran **Odia:** Ole

2.3 Biological Source:

Elephant Foot Yam is the tuber obtained from the plant Amorphophallus paeoniifolius (Dennst.) Nicolson, belonging to the family Araceae.

2.4 Geographical Source:

Distribution The plant is cultivated largely throughout India and also found wild from Punjab to West Bengal, Assam, Konkan, Dekkan, and Rampa Hills. It is also cultivated in Srilanka.

2.5 Scientific classification:

Kingdom: Plantae **Phylum:** Magnoliophyta **Order:** Alismatales **Family:** Araceae

Genus: Amorphophallus

Species: A. Paeoniifolius

Binomial name: Amorphophallus paeoniifolius (Dennst.) Nicolson.

2.6 Morphological Characteristics:

Plant: A thick, soft-stemmed herb with an underground round, flat, dark brown tuber (corm).

Leaves: Only one large leaf that is divided into smaller parts (leaflets).

Leaf stalk: Thick, spotted, and 60–90 cm long.

Leaflets: Oval or long, pointed, with many visible veins.

Flower: Male and female flowers grow close together on a single spike (spadix). No sterile (neuter) flowers present. The top part of the spadix is round or irregular in shape. A leaf-like cover (spathe) surrounds the flower, bell-shaped, greenish-pink outside and purple inside at the base, with wavy edges.

Fruits: Small, red, egg-shaped berries. Each fruit has 2–3 seeds

2.7 Traditional Uses:

1. Used to balance vitiated Vata and Kapha doshas.

2. Effective in treating joint pain (arthralgia).
3. Beneficial for elephantiasis and swelling disorders.
4. Traditionally applied in managing tumors (neoplasms).
5. Used for inflammatory conditions.
6. Helpful in haemorrhoids (piles) and bleeding disorders.
7. Treats vomiting (emesis).
8. Used in cough, bronchitis, and asthma as an expectorant.
9. Stimulates appetite and aids digestion treats anorexia, dyspepsia, and flatulence.
10. Relieves colic and constipation.
11. Acts as an anthelmintic to expel intestinal worms.
12. Supports liver health (hepatopathy) and spleen disorders (splenopathy).
13. Regulates menstrual disorders like amenorrhea and dysmenorrhea.
14. Enhances sexual vitality in cases of seminal weakness.
15. Used to combat fatigue, anemia, and general debility (Kumar et al., 2011).

3. LITERATURE REVIEW:

Nawaz et al. (2020) discuss how physical (e.g., heat-moisture, annealing) and chemical (e.g., oxidation, cross-linking) modifications change starch's structure and reactivity. These changes improve starch's functional properties like solubility, viscosity, and thermal stability, making it more suitable for food, pharmaceutical, and industrial uses. The paper highlights the role of starch source and the significance of tailored modifications for specific applications.

Krithika and Ratnamala (2019) review different starch modification techniques— physical, chemical, enzymatic, and genetic—used to improve starch properties for industrial use. Physical methods are eco-friendly, chemical ones enhance functionality but raise safety concerns, and enzymatic/genetic approaches offer precision. The paper emphasizes choosing methods based on application needs, cost, and environmental impact.

Neelam et al. (2012) review various techniques for starch modification—namely physical, chemical, and enzymatic methods—used to improve native starch's limitations such as low stability and solubility. They emphasize how these modifications enhance starch's functional properties, making it more suitable for diverse industrial applications, including food, pharmaceuticals, textiles, and biodegradable materials. The paper underlines the growing importance of modified starch as a sustainable alternative to synthetic materials.

Burrell (2003) presents a comprehensive analysis of the escalating necessity to enhance both the quality and quantity of starch in plant systems, particularly in light of the surging global demand for starch across food, feed, and industrial sectors. The manuscript elaborates on the biosynthetic pathways of starch within plants, accentuating the pivotal functions of essential enzymes, including ADP-glucose pyrophosphorylase (AGPase), starch synthases, branching enzymes, and debranching enzymes. Burrell elucidates how the genetic modification of these enzymatic components in agricultural species such as maize, potato, and rice can result in starches exhibiting altered structural properties, increased yield, or enhanced processing attributes. The review further explores the complexities associated with the modification of starch metabolism, encompassing regulatory intricacies and potential compromises regarding plant growth. In summary, the paper emphasizes

the significance of starch as a renewable resource and the transformative potential of biotechnological approaches to customize starch characteristics for specific applications.

Bertoft (2017) explores recent advancements in understanding the fine structure of starch, focusing on its two main components: amylose and amylopectin. The paper emphasizes that starch structure is more complex than previously thought, involving variations in chain length distribution, branching patterns, and the organization of crystalline and amorphous regions within starch granules. Bertoft discusses how new analytical techniques—such as enzymatic debranching, chromatography, and microscopy—have improved insights into the molecular architecture of starch. The review highlights how this structural knowledge is crucial for tailoring starch functionality in both food and industrial applications.

Stasiak et al. (2013) investigate the mechanical properties of native starch granules from maize, wheat, and potato using uniaxial compression tests. The study aims to understand how structural differences among starch types influence their physical behavior under mechanical stress. The authors report that potato starch granules exhibit the highest resistance to deformation, followed by wheat and maize, which is linked to differences in granule size, shape, and internal organization. This research provides valuable insights for food processing and material handling, where starch mechanical stability can affect texture and product performance.

Tharanathan (2005) reviews the various chemical, physical, and enzymatic modification techniques used to enhance the functional properties of starch for food and industrial applications. The paper explains how native starches have limitations such as poor solubility, instability under processing conditions, and retrogradation, which can be addressed through modifications like cross-linking, esterification, oxidation, and hydrothermal treatments. Tharanathan also discusses the importance of tailoring starch characteristics to meet specific requirements in processed foods, packaging, pharmaceuticals, and biodegradable materials. The review highlights the growing role of starch modification in adding commercial value and improving its utility across sectors.

Cornejo-Ramírez et al. (2018) provide a comprehensive review of the relationship between the structural characteristics of starches—such as granule morphology, amylose- to-amylopectin ratio, crystallinity, and molecular arrangement—and their functional properties, including gelatinization, pasting, swelling, and digestibility. The paper emphasizes how botanical origin influences starch structure, which in turn determines its behavior in food systems and industrial processes. It also covers how structural differences impact starch applications in terms of texture, stability, and nutritional value. The authors highlight the importance of understanding these relationships for the development of starch-based functional foods and improved processing techniques.

Gunaratne (2023) provides an in-depth overview of heat-moisture treatment (HMT) as a physical method for modifying starch properties without using chemicals. The chapter explains that HMT involves treating starch with limited moisture (typically 10–30%) at elevated temperatures (100–120°C), resulting in changes to crystallinity, granular structure, gelatinization behavior, and enzyme digestibility. Gunaratne emphasizes how HMT enhances starch thermal stability, reduces swelling, and alters pasting and rheological properties, making it suitable for various industrial and food applications. The review also discusses how starch origin and treatment conditions affect the outcome of HMT, highlighting its importance as a sustainable and clean-label modification technique.

Iqbal et al. (2024) investigate the impact of three physical modification techniques—heat moisture

treatment (HMT), annealing, and gelatinization-retrogradation—on the starch extracted from elephant foot yam (Amorphophallus paeoniifolius). The study reveals that these treatments significantly alter the starch's physicochemical, functional, and structural properties, such as swelling power, solubility, gelatinization temperature, crystallinity, and digestibility. Among the methods, HMT and annealing enhance thermal stability and reduce digestibility, while gelatinization-retrogradation leads to the formation of resistant starch. The paper highlights the potential of these eco-friendly treatments in customizing starch functionality for specific food and industrial uses.

Hong and Liu (2023) explore the pre-gelatinized modification of starch, a process where starch is partially cooked and then dried to enhance its solubility and ease of use in cold water. This modification disrupts the starch granules, making it ideal for instant foods and pharmaceutical applications due to its quick thickening ability and improved processing properties. The authors discuss various methods such as drum drying, extrusion, and spray drying, emphasizing how each affects the starch's structural and functional characteristics. They also highlight the influence of starch origin and processing conditions, while noting some drawbacks like loss of native granule structure and potential retrogradation.

The study by Suriya et al. (2019) explored how heat-moisture treatment (HMT) affects starch from elephant foot yam. They found that HMT improved the starch's thermal stability, gelatinization temperature, and resistance to digestion, while reducing its swelling and solubility. This suggests that modified yam starch could be useful in low- glycemic foods, packaging, and industrial applications. It adds new insights into using underutilized starch sources.

T. Raut (year not specified) examines the microscopical behavior of selected starches— such as corn, wheat, and potato—in combination with various food ingredients using Scanning Electron Microscopy (SEM). The study focuses on how starch granule morphology and surface structure are altered when starch is blended with sugars, fats, and proteins. SEM images reveal significant structural changes such as granule swelling, surface erosion, aggregation, and gelatinization depending on the type and concentration of the added ingredients. The paper highlights that these interactions influence starch's functional properties during food processing, including texture, water retention, and thermal behavior. The findings support the importance of microstructural analysis in optimizing starch-based food formulations.

Rashid et al. (2012) explore the gelatinization of starch using sodium silicate and analyze the physicochemical changes using a combination of techniques: Fourier Transform Infrared Spectroscopy (FTIR), Differential Scanning Calorimetry (DSC), X-ray Powder Diffraction (XRPD), and Nuclear Magnetic Resonance (NMR). The study reveals that sodium silicate significantly influences starch gelatinization by disrupting the crystalline regions and enhancing water uptake. DSC results show reduced gelatinization enthalpy, indicating structural destabilization, while XRPD and FTIR confirm the loss of crystallinity and changes in hydrogen bonding. NMR analysis further supports molecular reorganization. This multi-technique approach provides a deeper understanding of starch.

Pozo et al. (2018) investigate the structural order of native starch granules from different botanical sources using a combined approach of Fourier Transform Infrared Spectroscopy (FTIR) and X- ray Diffraction (XRD). The study focuses on evaluating the short- and long-range molecular order within starch granules to better understand their crystalline and amorphous domains. The authors find

that both FTIR and XRD effectively detect variations in crystallinity and molecular arrangement, influenced by starch origin and granule structure. The research highlights the complementary nature of these techniques in characterizing starch structure and underscores the importance of structural order in determining starch functionality, such as gelatinization, retrogradation, and mechanical behavior.

The paper by Lozano-Vazquez et al. (2021) investigates the properties of corn starch- calcium alginate xerogels using microscopy, thermal, X-ray diffraction (XRD), and Fourier Transform Infrared Spectroscopy (FTIR) analyses. It explores how the combination of corn starch and calcium alginate affects gel formation and its potential applications in food packaging, controlled drug delivery, and tissue engineering. The study emphasizes the importance of understanding the material's structure and stability for optimizing its use in various industries.

Apriyanto et al. (2022) present a comprehensive review of starch as a unique plant biopolymer, focusing on its molecular structure, biosynthesis, metabolism, and in plant modifications. The paper discusses the dual composition of starch (amylose and amylopectin), their structural complexity, and the enzymes involved in their synthesis and degradation. It highlights recent advances in genetic and metabolic engineering aimed at altering starch composition and architecture within the plant itself (in plants) to improve its functionality. The authors emphasize the potential of in plants modifications to tailor starch for specific industrial or nutritional applications without relying on post- harvest processing. The review also covers the regulation of starch metabolism and the integration of structural and functional studies for better utilization.

4. RATIONALE OF STUDY

Native starches from common sources like maize, potato, and tapioca often suffer from limitations such as poor solubility, low thermal stability, and weak flow properties, restricting their industrial applications. To overcome these drawbacks, there is a growing need for alternative, functional starch sources.

Amorphophallus paeoniifolius (Elephant Foot Yam) is an underutilized crop whose fruit tuber contains a significant amount of starch but remains largely unexplored. This study investigates the potential of starch extracted from the fruit tuber and enhances its functional properties through physical modification (pregelatinization) at controlled temperatures (62°C, 65°C, and 68°C). The goal is to develop a cost- effective, locally available starch with improved properties suitable for pharmaceutical, food, and industrial applications.

5. AIM AND OBJECTIVE

The aim of the present study is to extract, purify, thermally modify, and characterize starch obtained from the fruit tuber of Amorphophallus paeoniifolius (Elephant Foot Yam) in order to evaluate its physicochemical, structural, and functional properties for potential application in food, pharmaceutical, and industrial formulations.

- To achieve this aim, the following objectives were undertaken:
- To isolate native starch from the fruit tuber using wet extraction methods.
- To purify the extracted starch by removing non-starch impurities through Soxhlet extraction.
- To modify the purified starch using a pregelatinization process at controlled temperatures (62°C,

65°C, and 68°C).

- To evaluate the solubility of native, purified, and modified starches in various solvents.
- To assess the powder flow properties including bulk density, tapped density, angle of repose, Hausner's ratio, and Carr's index.
- To investigate the structural changes in starch samples using Fourier Transform Infrared Spectroscopy (FTIR) and X-ray Diffraction (XRD).

6. PLAN OF WORK

- Literature survey on starch.
- Procurement of chemicals.
- Isolation and purification and drying of native starch from elephant foot yam.
- Characterization of starches, and evaluating morphological, physicochemical and non- thermal treatment by using pressure techniques.
- Crystallinity of particle are evaluated by using X- ray diffraction (XRD).
- Spectroscopic characterization is carried out using physical methods like (UV – Visible, FTIR).
- Assessment of flow properties, water holding capacity and compressibility of native and modified starch.
- Statistical analysis and interpretation of data.

7. MATERIALS AND INSTRUMENTS

7.1 Materials:

- Fresh fruit tuber of Amorphophallus paeoniifolius (Elephant Foot Yam)
- Distilled water
- 0.1% Sodium metabisulfite solution
- 4% Sodium chloride (NaCl) solution
- Petroleum ether or hexane (for Soxhlet extraction)
- Ethanol
- Acetic acid
- Sulphuric acid
- Nitric acid
- Hydrochloric acid
- Propylene glycol

Glassware:

- Beakers (100 mL, 250 mL, 500 mL)
- Measuring cylinders (25 mL, 50 mL, 100 mL)
- Funnels
- Test tubes
- Petri dishes
- Round-bottom flask (for Soxhlet extraction)
- Filter paper or cellulose thimble

- Glass rods
- Watch glass

7.2 Instruments:

- Waring blender or homogenizer
- Centrifuge
- Hot air oven
- Analytical weighing balance
- Soxhlet apparatus with condenser
- Heating mantle or thermostatic water bath
- 100 µm sieve
- 60-mesh sieve
- Funnel stand (for angle of repose measurement)
- Stopwatch (for tapped density procedure)

8. EXPERIMENTAL WORK

8.1 ISOLATION OF STARCH:

The Elephant foot yam starch isolation process involves several steps to extract and purify the starch granules from the tubers. The following is a typical procedure used in isolating starch from Elephant Foot Yam:

Material Requirement:

- Fresh Elephant Foot Yam tuber.
- Distilled Water.
- 0.1% Sodium metabisulfite solution (optional).
- 4% NaCl (Sodium Chloride) solution.
- Warning blender or homogenizer.
- 100 um sieves.
- Centrifuge.
- Oven (For drying the starch).
- Filter Paper.
- Weighing balance.

Procedure:

- First, peel the elephant foot yam tuber to remove the skin.
- Then cut it into small pieces to increase surface area for efficient extraction of starch.
- These pieces immediately suspended in 0.1% sodium metabisulphite solution to prevent enzymatic reaction and oxidation.
- The yam pieces are homogenized using warning blender which breaks down tuber's structure releasing the starch granules.
- The homogenized sample suspended in large volume of 4% sodium chloride (NaCl) solution which helps in separating starch from protein and impurities.
- The slurry passed through 100um sieve to remove fiber and large particle allow only the finer starch contain liquid to pass through it.
- The filtrate is then centrifuged at 2660g for 15min.
- This process repeated four time to ensure maximum starch and purity.
- The final starch sediment which appears is oven dried for 8 hours at 45ºC.
- Grind the starch into fine powder Kaur et al., 2004.

Elephant foot yam fruit tuber. Peeled and cut pieces of tuber. Starch.

8.2 PURIFICATION OF STARCH:

The Elephant foot yam fruit starch that is isolated and dried is purified to remove impurities and

protein. The Soxhlet extraction method is primarily used to remove non- polar impurities such as lipids (fats and oils) from starch. After isolating starch from elephant foot yam, further purification using Soxhlet apparatus improves the purity by eliminating fat-soluble contaminants.

Material Required:

- Isolated elephant foot yam starch (dry).
- Soxhlet apparatus (including round-bottom flask, extraction chamber, condenser).
- Non-polar solvent (e.g., petroleum ether or hexane).
- Filter paper thimble (or cellulose thimble).
- Heating mantle or water bath.
- Oven (for drying starch).
- Weighing balance.

Procedure:

- The elephant foot yam fruit starch which is isolated from the tuber is oven dried at 40-50ºC to remove the moisture.
- Now, prepare the Soxhlet set-up.
- Weigh 10-20 gm of dry elephant foot yam starch.
- Place the weighed elephant foot yam starch into the filter paper or cellulose thimble.
- Now, pour 200-300 ml of petroleum ether or hexane into the round bottom flask.
- Assemble the Soxhlet apparatus (flask, extractor, condenser).
- Heat using a mantle or water bath at 60-70ºC.
- Solvents vapors condense and wash the starch repeatedly, extracting fats.
- Continue extraction for 4 to 6 hours or until solvent run clears in the siphon.
- Cool the setup and remove the thimble.
- Air-dry in a fume hood to evaporate residual solvent. Further oven-dry at 45°C for several hours.
- Grind to fine powder (if needed). Store in an airtight container for future use Heinze et al., 2001.

Fig.8.1: Extraction assembly of Elephant Foot Yam Starch

8.3 PREGELATINIZATION PROCESS OF STARCH:

The purified Elephant foot yam starch is further modified by using the pregelatinized process. In which the purified Elephant foot yam starch is heated at various temperature to pregelatinized the it for enhancing the starch properties. The pregelatinization process is as follows:

- Weigh 25 grams of purified starch and mix it with 100 milliliters of distilled water, maintaining a starch-to-water ratio of 1:3 to ensure proper gelatinization.
- Heat the mixture at different temperature such as, 62°C, 65°C, or 68°C while continuously stirring.
- Maintain the heating for 10 minutes to allow the starch to swell and form a gelatinous paste.
- Transfer the gelatinized paste into a drying oven and dry it at a constant temperature of 60°C for 8 hours.
- This step removes moisture and converts the paste into a solid, crisp form.
- After drying, grind the solidified starch paste into a fine powder using a suitable grinder or mill.
- Pass the powdered starch through a 60-mesh screen to obtain uniform particle size and remove any coarse particles Raut, n.d 2021.

Pregelatinized starch 62°C. Pregelatinized starch 65°C. Pregelatinized starch 68°C.

8.4 CHARACTERIZATION OF STARCHES:

8.4 Physicochemical Characterization:

a) Solubility: The solubility was determined in each of the following solvents such as - Cold water, hot water, Sulphuric acid, acetic acid, nitric acid, ethanol, HCL, propylene glycol.

b) Bulk Properties: The bulk properties describe the Physicochemical characteristics of powdered or granual materials, such as, starch in their aggregated form rather than at molecular level. These properties include bulk density, tapped density, compressibility. These properties reflect how starch behaves during storage, handling, and processing, which is essential in determining its suitability for various industrial applications. For example, compressibility is vital in tablet formation in pharmaceuticals. Understanding these properties helps in predicting the material's behavior under different mechanical and environmental conditions Jivraj, Martini and Thomson, 2000.

1) Bulk Density: To determine the bulk density procedure similar to that of a bulk densitometer was employed, as described by Obitte and Chukwu 2007. A 5 g quantity of starch powder was accurately weighed and transferred into a 25 ml graduated measuring cylinder. The top surface of the powder bed was gently leveled without compacting the sample, and the occupied volume was recorded. Bulk density is then calculated by using the following formula:

Bulk density (g/cm^3) = Weight of starch/ Bulk volume.

2) Tapped density: Tapped density was determined by taking the same 10 g of starch powder and tapping the measuring cylinder gently 150 times on a cushioned surface, as described by Obitte and Chukwu 2007. After tapping, the new (reduced) volume of the starch was recorded. The tapped density was then calculated using the formula:

Tapped density (g/cm^3) = Mass of starch / Tapped volume

This method helps to assess how well the starch particles settle or pack when vibration or handling occurs.

c) Powder Flow Properties: Powder flow properties refer to the ability of powdered substances, such as starch, to move or flow under specific conditions. These properties are crucial for processing, packaging, and handling in pharmaceutical, food, and cosmetic industries. Factors such as particle size, shape, surface texture, moisture content, and density influence how easily a powder flow. Poor flowability can lead to issues like inconsistent mixing, irregular dosing, and blockage in machinery. Therefore, evaluating flow properties helps ensure efficient production and product quality Prescott and Barnum, 2000.

1) Angle Of Repose: The angle of repose is defined as the maximum angle at which a material can be piled without slumping. It represents the steepest angle of descent relative to the horizontal plane to which a granular material can be stacked without collapsing under the force of gravity. This property is widely used to evaluate the flow characteristics of powders and granules in various industries, especially in food, pharmaceutical, and agricultural processing Shah et al., 2008.

Procedure:

- Fix the funnel vertically using a stand so that its tip is exactly 2 cm above the center of a Petri dish with a 9 cm diameter.
- Carefully pour the starch powder into the funnel, allowing it to fall freely through the tip without shaking or disturbing.
- Allow the powder to form a cone-shaped heap in the dish. Stop the flow once the top (apex) of the heap just reaches and touches the funnel tip.
- Use a ruler to measure the vertical height from the base (Petri dish) to the top of the powder cone.
- Measure the diameter of the base of the cone (i.e., how wide the bottom circle of the heap is).
- Divide the diameter by 2 to get the radius (r), which is needed for angle calculation.
- This formula relates the height and radius of the cone to the angle of repose.
- Use a calculator or scientific calculator to find the angle by taking the arctangent (inverse tangent) of h divided by r.
- This angle (θ) shows how well the powder flows; smaller angles mean better flow, larger angles indicate poor flow Aulton & Taylor, 2018.

Angle of repose	Type of flow
<25	Excellent
25-30	Good
30-40	Passable
>40	Very Poor

Table 8.4.1 Flow properties of powder depending upon angle of repose.

Fig.8.2: Funnel method (Angle of repose)

2) Hausner's Ratio:

Hausner's ratio is a simple but effective indicator used to assess the flowability of powders by comparing the degree of densification achieved through tapping. It is calculated as the ratio of the tapped density to the bulk density of a powder:

Hausner's Ratio = Tapped Density / Bulk Density

A value close to 1 (typically between 1.00 and 1.25) indicates good flowability and low inter particle friction. Higher values (>1.25) suggest increased cohesiveness, poor flow, and potential problems in manufacturing processes like tableting or capsule filling. Hausner's ratio is especially useful for evaluating excipients like starch in direct compression formulations, where flow behavior is critical for dosage uniformity Staniforth et al., 2007.

2. Carr's Compressibility Index: Carr's compressibility index, also known as Carr's Index, is a widely used parameter for determining the flow characteristics and compressibility of powdered substances. It measures the percentage difference between tapped and bulk densities, offering insight into how much a powder can consolidate under pressure:

Carr's Index (%) = [(Tapped Density – Bulk Density) × 100] / Tapped Density

Carr's Index reflects the extent of inter particle interactions. Lower index values (5–15%) indicate excellent flow properties and low compressibility, whereas higher values (>25%) are indicative of poor flow and a tendency for particles to bridge or clog equipment. This index is particularly relevant in industries where powder flow consistency affects production efficiency and product quality Staniforth et al., 2007.

Carr's index (%)	Type of flow
5-15	Excellent
12-16	Good
18-21	Fair to passable
23-35	poor
33-38	Very poor

Table 8.4.2 flow properties of powder

8.5 Physical Methods:

Physical characterization methods are essential analytical tools used to investigate the structural, morphological, and physicochemical properties of materials such as starch. These techniques provide crucial insights into parameters like crystallinity, molecular bonding, thermal behavior, particle size, and surface morphology, all of which influence the functionality and application of the material in industries such as pharmaceuticals, food, and cosmetics. Common physical techniques include Fourier Transform Infrared Spectroscopy (FTIR) for identifying functional groups, X-ray Diffraction (XRD) for crystalline structure analysis. These methods offer a non- destructive means to comprehensively understand the material's composition and performance under different conditions Aulton and Taylor, 2013.

a) Fourier Transform Infrared (FTIR) Spectroscopy:

FTIR spectroscopy is a widely used technique for characterizing the molecular structure and identifying functional groups in starch and other polysaccharides. In this study, the FTIR spectra of the starch samples were recorded in the wavenumber range of 4000 to 400 cm^{-1} using the potassium bromide (KBr) pellet method. The finely powdered starch was mixed with spectroscopic-grade KBr and compressed into a thin pellet, which was then scanned using a FTIR spectrophotometer. This method allows for the detection of specific vibrational modes corresponding to chemical bonds, such as O- H, C-H, and C-O stretching, providing insights into the degree of hydrogen bonding, branching, and crystallinity of starch Abdullah et al., 2018.

Importance: FTIR analysis is essential for understanding the chemical integrity and any modifications in starch structure due to physical or chemical treatment. It also helps identify potential interactions with other formulation components in pharmaceutical or food products.

b) X-ray Diffraction (XRD) Analysis:

X-ray diffraction is an effective technique for analyzing the crystalline structure of starch granules. In this study, X-ray diffraction patterns were obtained using a Rigaku Miniflex 600 diffractometer. The diffraction scans were performed over an angular range of 10° to 90° (2θ) at a voltage of 40 kV, providing detailed information on the arrangement of crystalline and amorphous regions within the starch. The relative crystallinity of the sample was calculated by determining the ratio of the area under the crystalline peaks to the total area under the curve, which includes both crystalline and amorphous contributions Ye, 2018.

Importance: XRD is crucial for evaluating the crystalline type (A, B, or C) of starch, which influences its gelatinization behavior, digestibility, and mechanical properties. A higher degree of crystallinity generally correlates with enhanced structural stability and resistance to enzymatic breakdown, making it a key factor in food and pharmaceutical applications.

9. RESULT AND DISCUSSION

9.1 Solubility Results:

9.1.1 Solubility of native starch:

Sr no	Solvent	Solubility
1	Cold H2O	Insoluble clear liquid with settled starch.
2	Hot H2O	Gelatinized or gel form /soluble.
3	Sulphuric Acid	Dark color possibly reacts not soluble.
4	Acetic Acid	Some residual present insoluble or partially

		soluble.
5	Nitric Acid	Some residue with color change partially soluble with reaction.
6	Ethanol	Clear separation &sedimentation indicating insoluble.
7	Propylene Glycol	--------
8	HCL	Slight residue partially soluble with reaction.

Native starch shows very limited solubility across all tested solvents due to its highly ordered crystalline and granular structure. In cold water, it remains insoluble and settles at the bottom without any visible swelling or dispersion. When exposed to hot water, it swells slightly, forming a turbid suspension but never dissolves completely due to the rigidity of its semi-crystalline granules. In organic solvents like ethanol and propylene glycol, and weak acids like acetic acid, native starch also shows poor solubility, typically forming sediment or clumps at the bottom of the container. Strong acids like sulfuric acid, nitric acid, and hydrochloric acid (HCl) cause minimal visible dispersion or reaction; sulfuric acid may cause partial hydrolysis, but the interaction is weak overall. These observations suggest that native starch has limited accessibility to solvents, both polar and acidic, without thermal or chemical treatment.

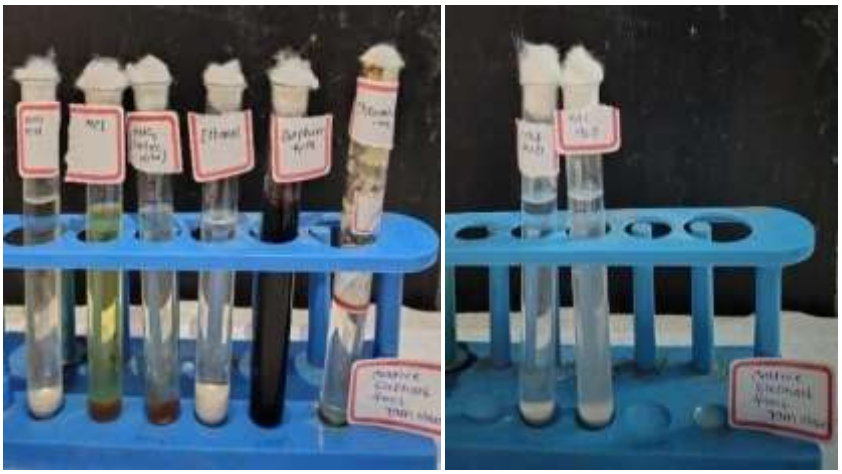

Fig 9.1.1 Solubility of Native Starch

9.1.2 Solubility of purified starch:

Sr no	Solvent	Solubility
1	Cold water	Slightly less clear suggest partial solubility.
2	Hot H2O	Clear solution good solubility.
3	Sulphuric Acid	Black dark color change implies chemical reaction not soluble.
4	Acetic Acid	Solution implies insoluble or poorly soluble (suspended particle visible).
5	Nitric Acid	Sample appear partially soluble or lightly colored suggested reaction.

6	Ethanol	------
7	Propylene Glycol	Sample is partially soluble.
8	HCL	Sample is clear or mostly insoluble indicate poor solubility.

After purification, the starch becomes slightly more reactive and dispersible. In cold water, the solubility remains poor, but in hot water, purified starch shows improved swelling and forms a more uniform suspension, though full clarity is still not achieved. Its interaction with ethanol and propylene glycol improves slightly, likely due to the removal of surface-bound lipids and proteins that hinder solvent contact. In acetic acid, a mild acid, purified starch shows better dispersion than native starch but still forms some residue. Strong acids like sulfuric acid, nitric acid, and HCl initiate more visible reactions with purified starch compared to the native form, with sulfuric acid causing partial breakdown. However, even after purification, the core structure remains too compact to allow full dissolution in any solvent.

Fig 9.1.2 Solubility of Purified starch

9.1.3 Modified starch at 62°C:

Sr no	Solvent	Solubility
1	Cold H2O	Turbid indicating limited solubility compared to hot H2O.
2	Hot H2O	Clear to slightly turbid indicating good solubility.
3	Sulphuric Acid	Appears dark and thick indicating chemical reaction not solubility.
4	Acetic Acid	Turbid suggesting poor solubility.
5	Nitric Acid	Slight turbidity indicating poor solubility & slight reaction.
6	Ethanol	Partially soluble.
7	Propylene Glycol	Solution suggests partially soluble.
8	HCL	Partially soluble.

Once the starch is modified thermally, beginning at 62°C, significant changes in solubility are

observed. In hot water, modified starch starts to swell more effectively, forming a moderately turbid but more uniform solution. In cold water, slight dispersion may begin to appear. With ethanol and propylene glycol, there's some improvement in dispersion, although a degree of sedimentation still occurs. In acetic acid, the starch shows moderate swelling and better interaction compared to the purified form. In sulfuric acid, a clearer reaction begins to emerge as hydrolysis increases. Similarly, nitric acid and HCl produce more homogeneous suspensions than the purified.

Fig 9.1.3 Solubility of Modified starch at 62°C

9.1.4 Modified Starch at 65°C:

Sr no	Solvent	Solubility
1	Cold H2O	Slight cloudiness slight soluble.
2	Hot H2O	Clear dispersion soluble.
3	Sulphuric Acid	Cloudy with sediment and reaction occurs.
4	Acetic Acid	Partially Soluble.
5	Nitric Acid	Yellow brown solution likely reaction.
6	Ethanol	Partially soluble.
7	Propylene Glycol	Clear dispersion soluble.
8	HCL	Clear with sediment slight soluble.

At 65°C, solubility reaches a favorable point. In hot water, the starch now shows a near- clear dispersion, indicating advanced gelatinization. Cold water shows better swelling. Most notably, in ethanol, the starch becomes well-dispersed and visibly soluble, with significantly reduced sedimentation—this represents an optimal interaction between the moderately polar ethanol and the now partially amorphous starch. Other solvents like propylene glycol, acetic acid, and strong acids also show clearer and more stable dispersions, suggesting increased solvent accessibility.

Fig 9.1.4 Solubility of Modified at 65°C

9.1.5 Modified starch at 68°C:

Sr no	Solvent	Solubility
1	Cold H2O	Slightly soluble.
2	Hot H2O	Very soluble.
3	Sulphuric Acid	Highly reactive and partially decomposed.
4	Acetic Acid	Partially soluble.
5	Nitric Acid	Turbid yellow solution & reaction.
6	Ethanol	Visible sediment.
7	Propylene Glycol	Soluble.
8	HCL	Partially soluble with moderate cloudiness.

Surprisingly, at 68°C, despite more extensive gelatinization, the solubility in ethanol decreases compared to 65°C. The data shows visible sedimentation reappearing, which may be due to over-gelatinization or molecular aggregation at higher temperatures. This can cause retrogradation or re-association of starch chains, especially in solvents like ethanol where hydrogen bonding with the solvent is limited. In contrast, solubility in hot water reaches its maximum, forming a fully homogenous solution. Cold water shows noticeable swelling. Acetic acid, propylene glycol, and strong acids like sulfuric acid, nitric acid, and HCl continue to show enhanced interactions and breakdown of starch granules.

Fig 9.1.5 Solubility of Modified at 68°C

9.2 Bulk properties and powder flow properties:

Starch	Bulk density	Tapped density	Angle of repose	Hausner's Ratio	Carr's index
Native starch	0.6097	0.7812	0.62	1.2812	21.953
Purified starch	0.6024	0.7692	0.71	1.2768	21.684
Modified starch at 62°C	0.7692	0.9615	0.71	1.25	20
Modified starch at 65°C	0.7986	0.9803	0.75	1.2352	19.045
Modified starch at 68°C	0.8233	0.9259	0.76	1.1111	10.001

Table 9.2.1 Bulk and Powder flow Properties

The native starch shows a bulk density of 0.6097 g/cm³ and a tapped density of 0.7812 g/cm³, which slightly improves in the purified starch (0.6024 g/cm³ and 0.7692 g/cm³, respectively). However, these two types of starches both display relatively high Hausner's ratios (1.2812 and 1.2768) and Carr's indices (21.953% and 21.684%), indicating poor to fair flow properties. Additionally, their angles of repose (0.62 for native and 0.71 for purified) suggest a moderate tendency toward cohesive behavior.

Upon modification at 62°C, the starch shows a marked improvement in density, with a bulk density of 0.7692 g/cm³ and a tapped density of 0.9615 g/cm³. This suggests a tighter particle packing ability compared to native and purified forms. The Hausner's ratio drops to 1.25 and Carr's index to 20%, showing improved but still marginal flowability. The angle of repose remains at 0.71, similar to the purified starch.

Modification at 65°C further enhances density (bulk: 0.7986 g/cm³, tapped: 0.9803 g/cm³), and continues the trend of improving flow. The Hausner's ratio drops to 1.2352 and Carr's index to 19.045%, both suggesting better flow characteristics than the 62°C- modified starch. However, the angle of repose increases slightly to 0.75, indicating a slight decrease in flow based on this parameter alone.

Starch modified at 68°C exhibits the most favorable characteristics. It has the highest bulk (0.8233

g/cm³) and tapped (0.9259 g/cm³) densities, reflecting excellent packing

potential. Most notably, it shows the lowest Hausner's ratio (1.1111) and Carr's index (10.001%), which are indicative of excellent flow properties. Despite having the highest angle of repose (0.76) among all samples, which might suggest some cohesiveness, the overall interpretation based on all parameters confirms this as the best-performing starch in terms of flowability and compressibility.

9.3 X-Ray Diffraction:

a) XRD of Native starch:

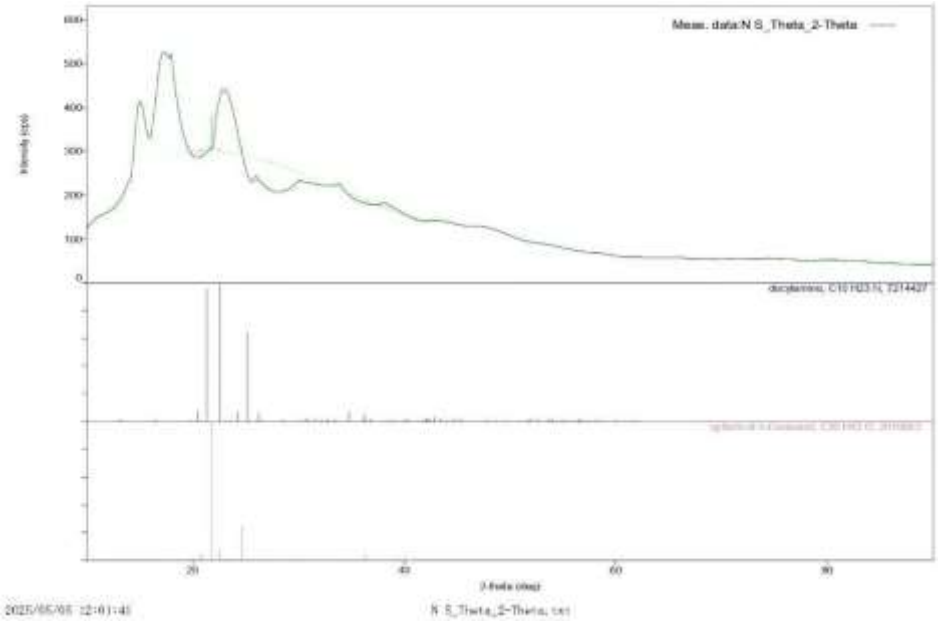

Fig 9.3.1 XRD of Native starch

No.	2-theta	d	FWHM	Int. I	Size
	(deg)	(ang.)	(deg)	(cps deg)	(ang.)
107	23.10(7)	3.847(11)	1.39(18)	175(26)	61(8)
108	71.0(3)	1.327(5)	3.4(11)	7(4)	30(10)
109	74.9(6)	1.266(8)	6.6(6)	63(5)	15.7(15)
110	80.84(16)	1.1880(19)	5.1(5)	32(4)	21.2(19)

Table 9.3.1 XRD readings of Native starch

The native starch exhibited strong and sharp diffraction peaks located at approximately 15.1°, 17.3°, 20.2°, and 23.1° (2θ). These peaks are characteristic of an A-type crystalline structure, which is typically associated with tightly packed double helices of amylopectin in tuber and cereal starches. The intensity of these peaks reflects a high degree of molecular order within the starch granules, indicating that the starch in its natural form possesses a semi-crystalline structure, where crystalline and amorphous zones coexist in a well-balanced manner. This semi-crystalline nature contributes to the granules' natural resistance to swelling and enzymatic degradation Zeng et al.,2015.

b) XRD of Purified starch:

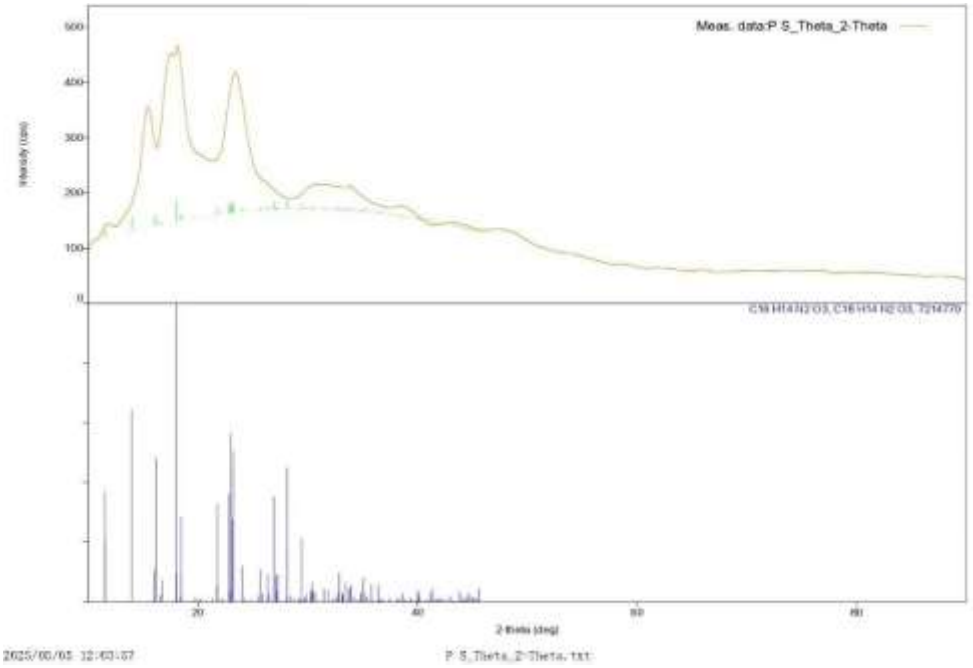

Fig. 9.3.2. XRD of Purified starch

No.	2-theta	d	FWHM	Int. I	Size
	(deg)	(ang.)	(deg)	(cps deg)	(ang.)
111	18.20(3)	4.870(9)	4.04(5)	1055(14)	20.8(3)
112	23.38(6)	3.802(9)	2.04(6)	443(9)	41.5(13)
113	31.79(10)	2.813(9)	5.9(3)	166(10)	14.6(8)
114	71.7(7)	1.315(12)	9.9(9)	74(4)	10.4(9)
115	76.5(3)	1.244(4)	5.2(9)	28(4)	20(4)
116	80.4(3)	1.193(4)	3.2(11)	15(3)	34(11)

Table 9.3.2 XRD readings of Purified starch

Upon purification, the XRD pattern maintained the same major peaks at 15.1°, 17.2°, 20.1°, and 23.0° (2θ) but the peaks appeared sharper and more defined than those of the native starch. This suggests that while the fundamental A-type crystalline structure remained intact, the removal of non-starch materials such as proteins, lipids, and other impurities resulted in a more ordered internal arrangement. The enhanced peak intensity is indicative of a greater proportion of crystalline regions relative to amorphous content. Thus, purification improved the molecular organization within the starch granules without altering the inherent crystal type Hoover et al.,2010

c) XRD of Modified starch at 62°C:

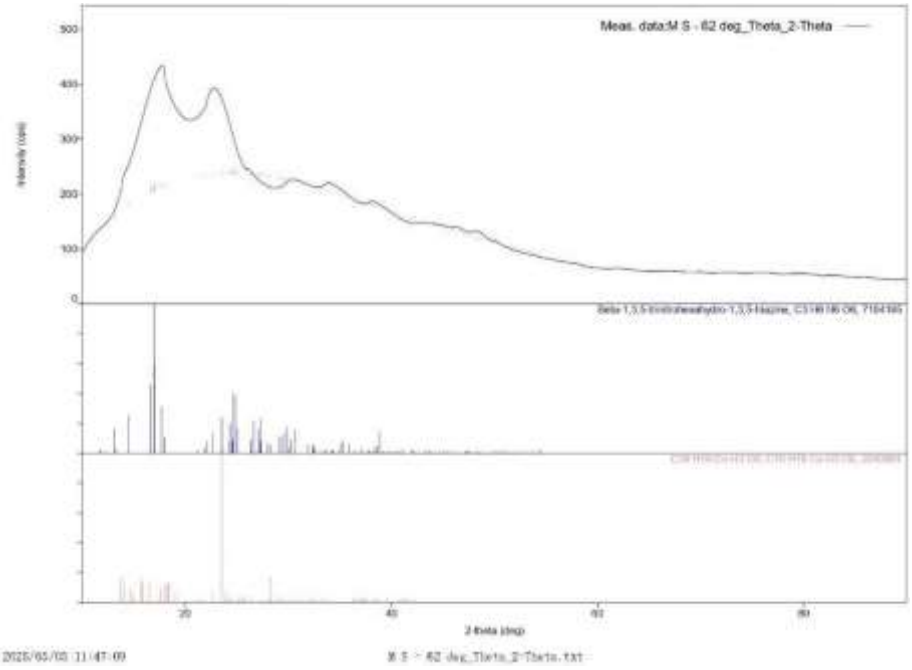

Fig. 9.3.3.XRD of Modified starch at 62°C

No.	2-theta (deg)	d (ang.)	FWHM (deg)	Int. I (cps deg)	Size (ang.)
96	22.65(7)	3.923(11)	4.5(2)	884(25)	18.8(10)
97	71.0(9)	1.326(15)	8.6(9)	55(7)	11.8(12)
98	75.7(4)	1.255(6)	7.4(13)	34(6)	14(2)
99	80.12(16)	1.197(2)	9.5(4)	86(5)	11.4(5)

Table 9.3.3 XRD readings of Modified starch at 62°C

When the starch was thermally modified at 62°C, noticeable changes in the diffraction pattern were observed. The peaks that were prominent in the native and purified forms became weaker in intensity, and some new minor peaks emerged. Specifically, the peaks around 15.2°, 17.4°, 19.8°, and 22.9° (2θ) were still visible, but much less intense. This indicates that the crystalline regions began to break down due to the thermal energy, although a part of the original structure remained Liu et al.,2014. The presence of both reduced crystalline peaks and additional signals suggests that the starch was in a transition state partially crystalline and partially amorphous. This is consistent with the early stages of gelatinization, where double helices within the crystalline lamellae start to unravel but are not yet fully disordered.

d) XRD of Modified starch at 65°C:

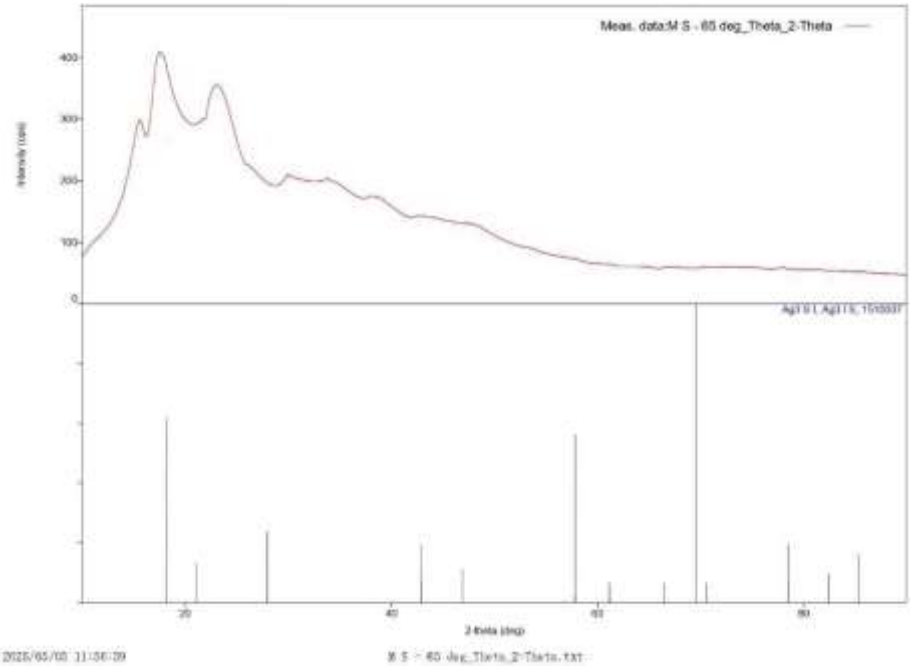

Fig. 9.3.4. XRD of Modified starch at 65°C

No.	2-theta	d	FWHM	Int. I	Size
	(deg)	(ang.)	(deg)	(cps deg)	(ang.)
100	72.7(8)	1.300(12)	5.1(10)	21(5)	20(4)
101	79.41(19)	1.206(2)	10.9(6)	112(10)	9.9(5)

Table 9.3.4 XRD readings of Modified starch at 65°C

As the modification temperature increased to 65°C, the diffraction pattern underwent further degradation. The peaks were much broader and lower in intensity, with diffuse features centered around the 17°, 20°, and 23° regions. This broadening reflects a semi- amorphous structure, where long-range molecular order has been largely lost. The thermal treatment at this stage appears to have disrupted most of the crystalline zones, leaving behind a structure with significantly higher molecular mobility. The starch granules are now largely gelatinized, resulting in properties such as higher solubility and altered pasting behavior.

e) XRD of Modified starch at 68°C:

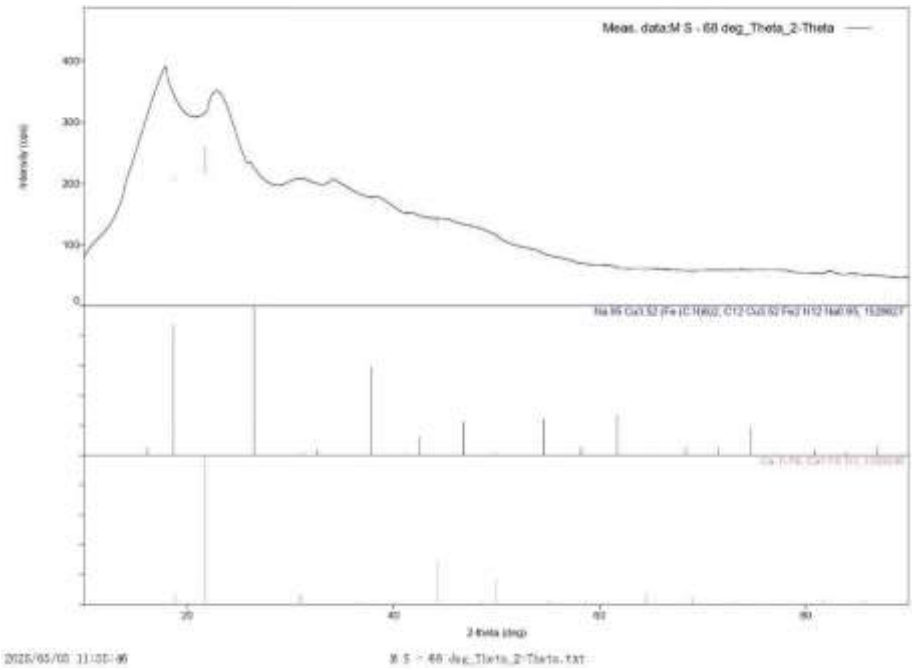

Fig. 9.3.5. XRD of Modified starch at 68°C

No.	2-theta (deg)	d (ang.)	FWHM (deg)	Int. I (cps deg)	Size (ang.)
102	22.91(7)	3.879(11)	4.4(3)	722(22)	19.5(11)
103	39.4(6)	2.29(4)	7.9(9)	204(17)	11.1(13)
104	71.9(4)	1.312(6)	5.5(11)	18(5)	19(4)
105	75.7(6)	1.256(8)	7.1(6)	62(6)	14.7(12)
106	81.8(5)	1.177(6)	9.6(5)	108(9)	11.5(5)

Table 9.3.5 XRD readings of Modified starch at 68°C

At the highest treatment temperature of 68°C, the diffraction pattern changed dramatically. The sharp peaks seen in the previous samples disappeared entirely, and only a broad, low-intensity hump was observed in the range of 15° to 25° (2θ). This lack of defined peaks is a clear indication of an amorphous structure, where the starch molecules no longer exhibit any regular arrangement. The original A-type crystalline form has been completely destroyed, likely due to extensive gelatinization and breakdown of the internal hydrogen bonds that once maintained the ordered regions. So, the modification at 68°C as compare to other converts the crystalline structure to the amorphous structure.

9.4 Fourier transform infrared spectroscopy:

a) FIR of Native starch:

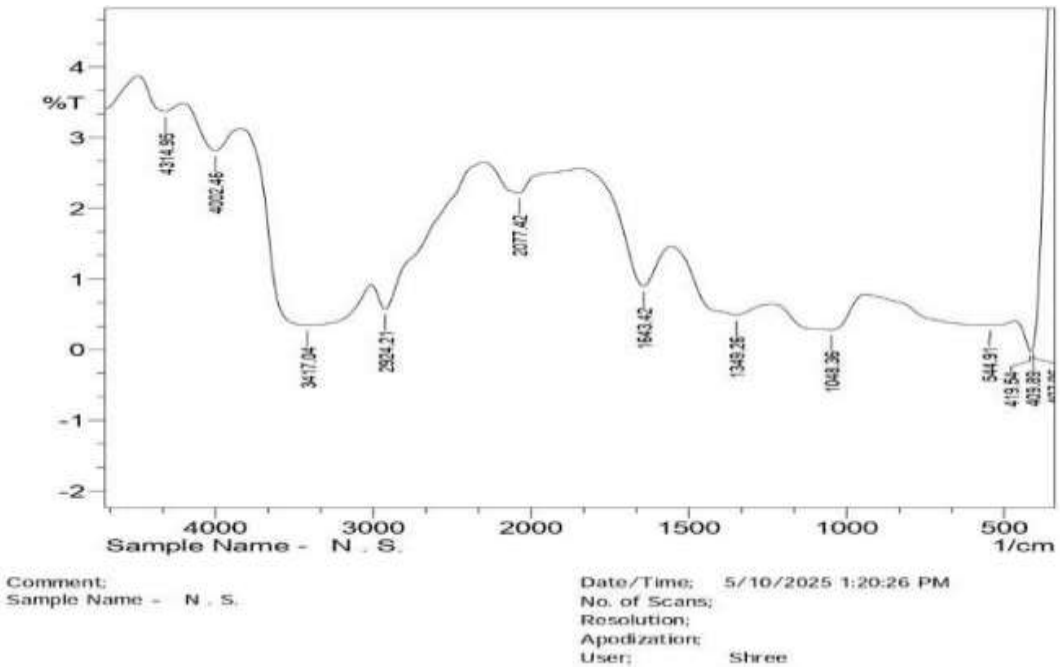

Figure.9.4.1 FTIR spectra of Native starch

SHIMADZU

No.	Peak	Intensity	Corr. Inte	Base (H)	Base (L)	Area	Corr. Are
1	407.96	0.015	0.105	408.93	339.49	119.937	0
2	409.89	0	0.002	410.86	409.89	4.566	0
3	419.54	0	-0.007	420.5	418.57	385.78	93.934
4	544.91	0.348	0.118	940.34	465.83	1105.446	37.513
5	1048.36	0.283	0.448	1233.53	941.3	695.693	67.672
6	1349.26	0.491	0.444	1554.69	1234.5	690.211	45.515
7	1643.42	0.898	0.895	1844.03	1555.66	516.768	24.426
8	2077.42	2.218	0.391	2300.21	1844.99	735.4	14.398
9	2924.21	0.573	0.558	3011.98	2301.18	1301.757	29.397
10	3417.04	0.346	1.659	3832.72	3012.94	1772.636	321.55
11	4002.46	2.817	0.471	4204.03	3833.69	560.369	11.654
12	4314.95	3.372	0.262	4489.51	4205	413.693	5.445

Sr.no.	Wavelength cm^{-1}	Peak observed	Vibration mode	Functional group
1	3412.38	4314.95	O–H stretching	Hydroxyl (–OH), Hydrogen bonding
2	2931.15	4288.92	C–H stretching	Aliphatic $–CH_2$
3	1643.55	4258.59	H–O–H bending	Absorbed water
4	1154.93	4244.59	C–O–C asymmetric stretching	Glycosidic linkage
5	1076.49	4234.71	C–O Stretching	Glucopyranose ring
6	1016.79	4226.61	C–O/C–C stretching	Crystalline/amorphous region

7	929.39	4217.70	Skeletal ring vibrations	Glucose unit
8	858.56	4209.42	C–H bending	Helical conformation
9	757.61	4200.73	C–Hout-of-plane bending	Fingerprint region

Table 9.4.1 FTIR readings of Native starch

The FTIR spectrum of the native elephant foot yam starch shows characteristic features of unmodified starch. A strong and broad O–H stretching vibration at 3412.38 cm^{-1} with the highest intensity of 4314.95 indicates extensive hydrogen bonding reflecting the semi- crystalline structure of native starch Kizil et al., 2002. The C–H stretching at 2931.15 cm^{-1} (4288.92) confirms the presence of –CH groups in the glucopyranose units Wang et al., 2015. The water bending peak at 1643.55 cm^{-1} (4258.59) reflects moderate water- holding capacity, typical of native starch with hydrophilic character Hoover, 2001. In the fingerprint region, the peaks at 1154.93 cm^{-1} (4244.59) and 1076.49 cm^{-1} (4234.71) correspond to C–O–C and C–O stretching vibrations, indicating the integrity of glycosidic linkages Zobel, 1988. The strong peak at 1016.79 cm^{-1} (4226.61) highlights the crystalline-amorphous balance of native starch Wang et al., 2015. Lower peaks at 929.39, 858.56, and 757.61 cm^{-1} confirm ring vibrations and helical structure, essential features of native starch granules van Soest et al., 1995.

b) FTIR of Purified starch:

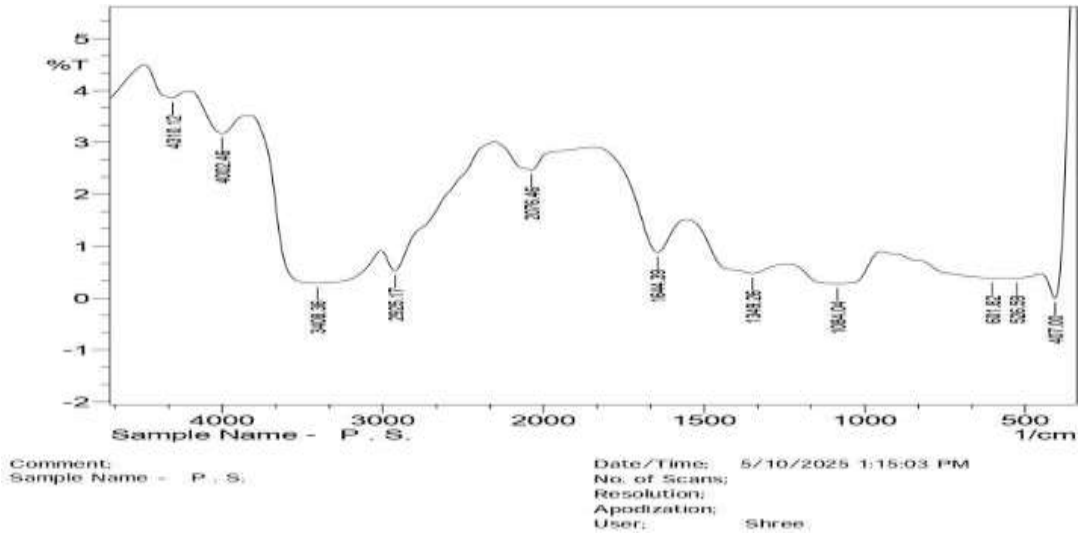

Figure.9.4.2 FTIR spectra of Purified starch

SHIMADZU

No.	Peak	Intensity	Corr. Inte	Base (H)	Base (L)	Area	Corr. Are
1	407	0.001	3.712	450.4	339.49	246.476	59.766
2	526.59	0.38	0.032	562.27	451.36	265.571	2.493
3	601.82	0.38	0.058	948.05	563.24	869.568	13.25
4	1084.04	0.275	0.501	1233.53	949.02	679.958	77.177
5	1349.26	0.473	0.486	1554.69	1234.5	689.859	48.309
6	1644.39	0.881	1.067	1842.1	1555.66	505.75	27.278
7	2076.46	2.473	0.487	2305.03	1843.06	722.242	15.967
8	2925.17	0.526	0.639	3011.01	2306	1275.332	32.725
9	3408.36	0.283	1.896	3825.97	3011.98	1795.582	373.934
10	4002.46	3.176	0.561	4205.96	3826.94	552.91	12.279
11	4310.12	3.855	0.322	4485.65	4206.93	388.58	5.86

Sr.no.	Wavelength cm^{-1}	Peak observed	Vibration mode	Functional group
1	3412.38	4312.38	O–H stretching	Hydroxyl (–OH), Hydrogen bonding
2	2931.15	4285.88	C–H stretching	Aliphatic $–CH_2$
3	1643.55	4253.94	H–O–H bending	Absorbed water
4	1154.93	4239.55	C–O–C asymmetric stretching	Glycosidic linkage
5	1076.49	4229.98	C–O stretching	Glucopyranose ring
6	1016.79	4221.42	C–O/C–C stretching	Crystalline/amorphous region
7	929.39	4212.05	Skeletal ring vibrations	Glucose unit
8	858.56	4203.38	C–H bending	Helical conformation
9	757.61	4194.72	C–H out-of-plane bending	Fingerprint region

Table 9.4.2 FTIR readings of Purified starch

The purified starch shows very similar peaks to the native sample, but with slightly reduced intensities e.g., the O–H stretch drops to 4312.38, and others follow a similar pattern. This reflects removal of impurities such as proteins and lipids, resulting in cleaner, sharper peaks Kizil et al., 2002. However, no major structural change occurs, as shown by the consistent positions of the main bands (C–O, C–H, and glycosidic regions). The reduction in water bending peak intensity at 1643.55 cm^{-1} (4253.94) suggests slightly less water-binding after purification Hoover, 2001.

c) FTIR of Modified starch at 62°C:

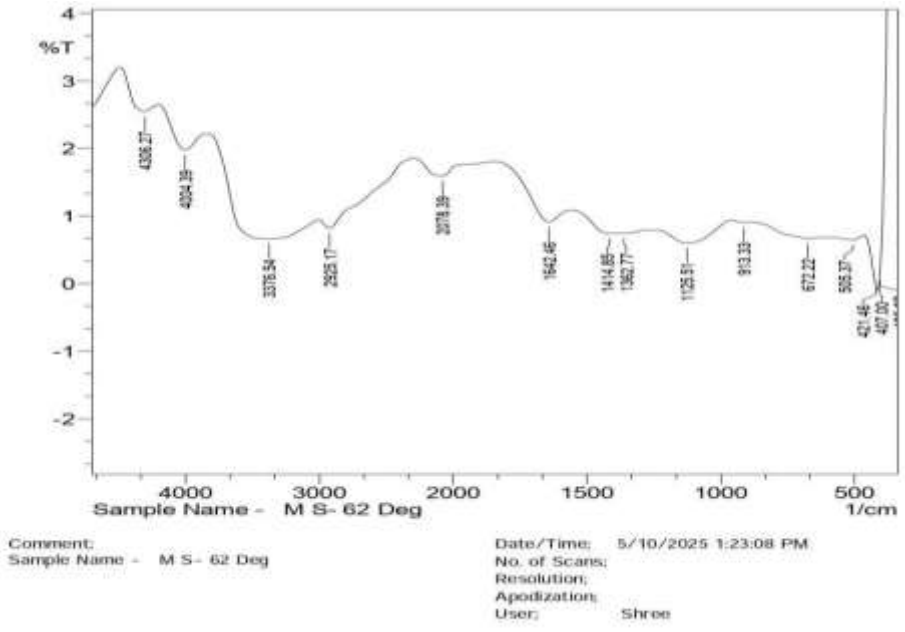

Figure.9.4.3 FTIR spectra of Modified starch at62°C

SHIMADZU

No.	Peak	Intensity	Corr. Inte	Base (H)	Base (L)	Area	Corr. Are
1	405.07	0.068	0.191	406.03	339.49	95.179	0
2	407	0	0.029	407.96	407	3.405	0
3	421.46	0	-0.019	422.43	420.5	385.78	93.926
4	505.37	0.645	0.061	593.14	465.83	276.791	2.426
5	672.22	0.668	0.077	884.4	594.1	617.309	6.928
6	913.33	0.902	0.015	960.59	885.36	153.467	0.3
7	1125.51	0.604	0.254	1257.64	961.56	634.549	23.293
8	1362.77	0.745	0.007	1379.16	1258.61	255.03	0.341
9	1414.85	0.742	0.07	1558.55	1380.13	367.371	3.072
10	1642.46	0.913	0.385	1842.1	1559.51	535.974	13.492
11	2078.39	1.594	0.239	2296.35	1843.06	800.322	12.853
12	2925.17	0.821	0.23	3009.08	2297.32	1351.573	16.91
13	3376.54	0.66	0.852	3831.76	3010.05	1677.278	166.009
14	4004.39	1.979	0.438	4202.11	3832.72	612.094	15.255
15	4306.27	2.546	0.298	4490.48	4203.07	449.957	8.446

Sr.no.	**Wavelength cm^{-1}**	**Peak observed**	**Vibration mode**	**Functional group**
1	3412.38	4310.10	O–H stretching	Hydroxyl (–OH), Hydrogen bonding
2	2931.15	4283.07	C–H stretching	Aliphatic $–CH_2$
3	1643.55	4250.68	H–O–H bending	Absorbed water
4	1154.93	4235.73	C–O–C asymmetric stretching	Glycosidic linkage
5	1076.49	4225.68	C–O stretching	Glucopyranose ring
6	1016.79	4217.29	C–O/C–C stretching	Crystalline/amorphous region
7	929.39	4208.38	Skeletal ring vibrations	Glucose unit
8	858.56	4199.70	C–H bending	Helical conformation
9	757.61	4190.52	C–H out-of-plane bending	Fingerprint region

Table 9.4.3 FTIR readings of Modified starch62°C

In 62°C, early signs of modification are observed. The O–H stretching intensity reduces further to 4310.10, indicating the beginning of hydrogen bond disruption or substitution of –OH groups van Soest et al., 1995. The water peak at 1643.55 cm^{-1} shows reduced intensity (4250.68), implying decreased moisture affinity. A small shift and broadening in the 1016.79 cm^{-1} region (4217.29) suggest a slight loss of crystallinity, moving toward a more amorphous structure. The skeletal ring vibrations (929.39, 858.56, 757.61 cm^{-1}) begin to weaken, indicating early molecular rearrangements Wang et al., 2015.

d) FTIR of Modified starch at 65°C:

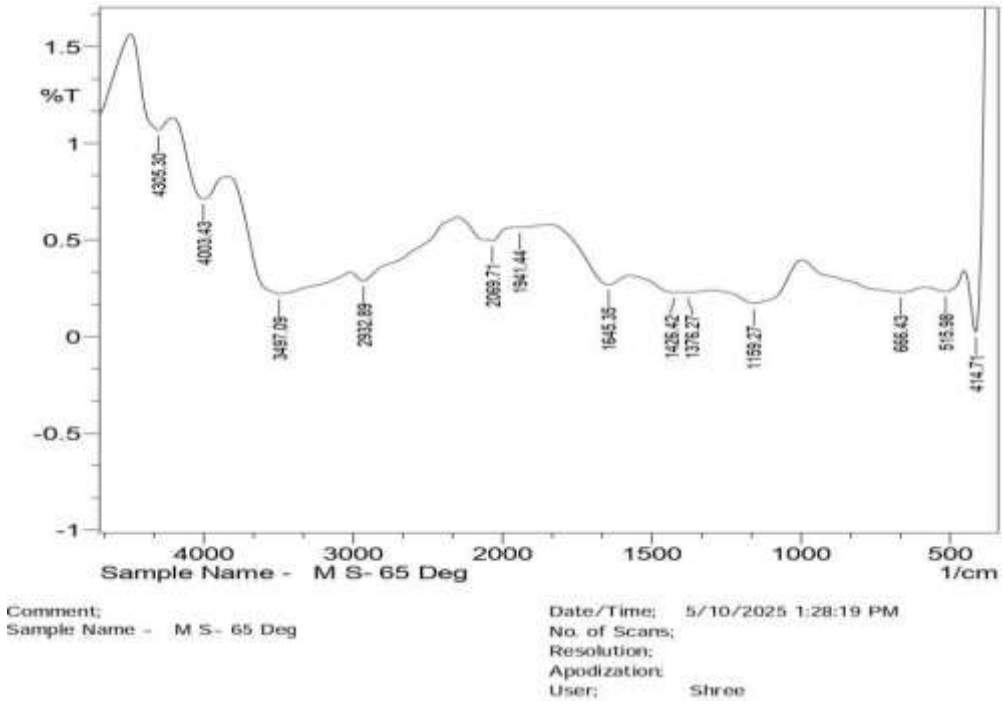

Figure.9.4.4. FTIR spectra of Modified starch at 65°C

SHIMADZU

No.	Peak	Intensity	Corr. Inte	Base (H)	Base (L)	Area	Corr. Are
1	414.71	0.025	2.448	452.33	339.49	245.805	43.399
2	515.98	0.235	0.066	583.49	453.29	337.529	8.419
3	666.43	0.231	0.054	996.28	584.46	1054.062	26.112
4	1159.27	0.173	0.14	1301.04	997.24	802.793	40.215
5	1376.27	0.229	0.004	1410.02	1302.01	284.351	0.445
6	1426.42	0.23	0.008	1573.02	1410.99	416.257	1.119
7	1645.35	0.269	0.117	1844.03	1573.98	651.206	13.283
8	1941.44	0.566	0.003	1958.8	1844.99	255.203	0.136
9	2069.71	0.498	0.086	2300.21	1959.76	771.718	13.535
10	2932.89	0.29	0.079	3020.66	2301.18	1710.155	26.256
11	3497.09	0.223	0.401	3831.76	3021.62	2022.785	176.435
12	4003.43	0.714	0.253	4207.89	3832.72	778.544	23
13	4305.3	1.069	0.211	4491.44	4208.86	542.979	12.965

Sr.no.	Wavelength cm^{-1}	Peak observed	Vibration mode	Functional group
1	3412.38	4307.82	O–H stretching	Hydroxyl (–OH), Hydrogen bonding
2	2931.15	4279.74	C–H stretching	Aliphatic $–CH_2$
3	1643.55	4246.52	H–O–H bending	Absorbed water
4	1154.93	4231.36	C–O–C asymmetric	Glycosidic linkage

			stretching	
5	1076.49	4220.43	C–O stretching	Glucopyranose ring
6	1016.79	4211.40	C–O/C–C stretching	Crystalline/amorphous region
7	929.39	4202.52	Skeletal ring vibrations	Glucose unit
8	858.56	4193.82	C–H bending	Helical conformation
9	757.61	4184.84	C–H out-of-plane bending	Fingerprint region

Table 9.4.4 FTIR readings of Modified starch65°C

The FTIR spectrum at 65°Creveals significant structural changes. The O–H peak further drops to 4307.82, reflecting a stronger breakdown of hydrogen bonds Kizil et al., 2002. Intensities across C–O and water peaks also decline, such as 1643.55 cm^{-1} (4246.52). The C–O–C and C–O peaks (1154.93–1016.79 cm^{-1}) become broader and weaker, especially at 1016.79 cm^{-1} (4211.40), suggesting reduction in crystalline domains and increased amorphous content van Soest et al., 1995. The lower region peaks (929.39 to 757.61 cm^{-1}) also diminish, showing degradation of ring structures and loss of helical order van Soest et al., 1995.

e) FTIR of Modified starch at 68°C:

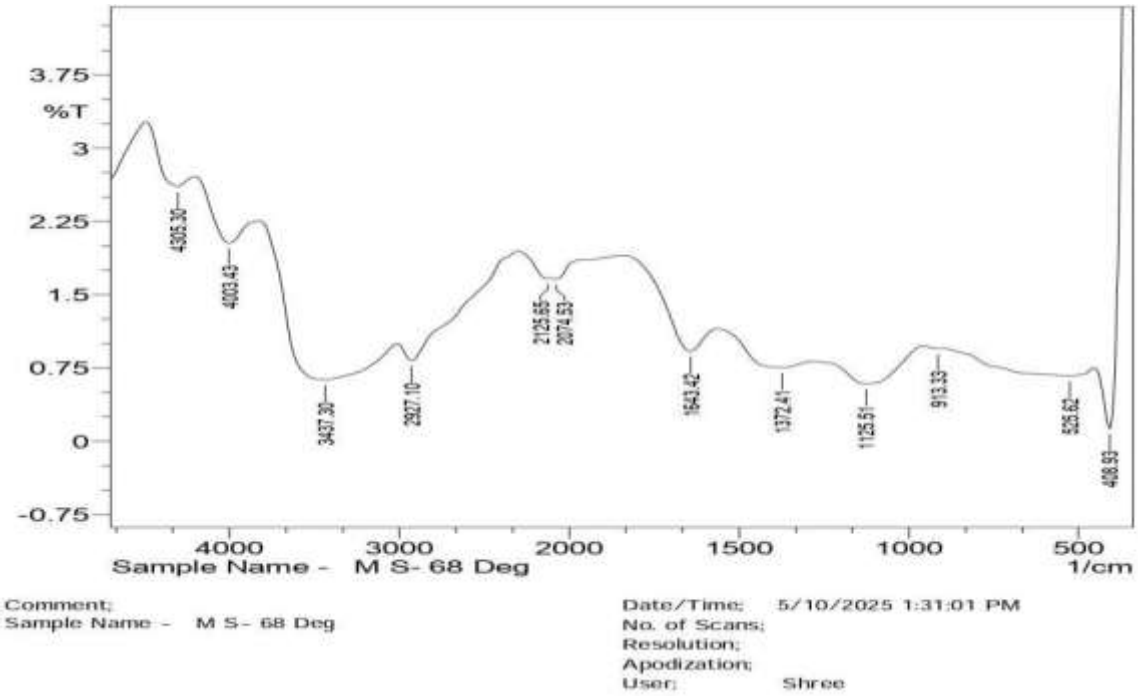

Figure. 9.4.5. FTIR spectra of Modified starch at 68°C

SHIMADZU

No.	Peak	Intensity	Corr. Inte	Base (H)	Base (L)	Area	Corr. Are
1	408.93	0.132	4.869	454.26	339.49	218.792	44.091
2	525.62	0.671	0.112	896.94	455.22	938.243	22.321
3	913.33	0.947	0.009	956.73	897.9	118.791	0.142
4	1125.51	0.59	0.303	1280.79	957.7	690.28	28.305
5	1372.41	0.754	0.171	1562.41	1281.75	578.982	14.084
6	1643.42	0.92	0.448	1840.17	1563.37	519.917	14.708
7	2074.53	1.66	0.04	2113.11	1841.13	474.78	0.612
8	2125.65	1.667	0.018	2300.21	2114.07	324.817	0.617
9	2927.1	0.826	0.287	3013.9	2301.18	1343.36	22.495
10	3437.3	0.633	1.024	3822.11	3014.87	1652.917	180.775
11	4003.43	2.029	0.443	4202.11	3823.08	624.262	15.198
12	4305.3	2.608	0.299	4490.48	4203.07	447.016	8.289

Sr.no.	Wavelength cm^{-1}	Peak observed	Vibration mode	Functional group
1	3412.38	4305.53	O–H stretching	Hydroxyl (–OH), Hydrogen bonding
2	2931.15	4277.32	C–H stretching	Aliphatic $–CH_2$
3	1643.55	4243.48	H–O–H bending	Absorbed water
4	1154.93	4227.84	C–O–C asymmetric stretching	Glycosidic linkage
5	1076.49	4216.10	C–O stretching	Glucopyranose ring
6	1016.79	4207.31	C–O/C–C stretching	Crystalline/amorphous region
7	929.39	4198.42	Skeletal ring vibrations	Glucose unit
8	858.56	858.56	C–H bending	Helical conformation
9	757.61	4180.84	C–H out-of-plane bending	Fingerprint region

Table 9.4.5 FTIR readings of Modified starch68°C

At 68°C exhibits the highest degree of modification. The O–H stretching intensity is lowest at 4305.53, indicating extensive alteration or substitution of hydroxyl groups, likely due to chemical or thermal modification Wang et al., 2015. The water-related band at 1643.55 cm^{-1} (4243.48) is significantly weaker, implying minimal water retention capacity Hoover, 2001. Sharp decreases and shifts in the C–O and glycosidic regions (especially 1016.79 cm^{-1} at 4207.31) confirm that the structure has become largely amorphous. Peaks in the 929–757 cm^{-1} range are greatly weakened or nearly disappeared, indicating substantial conformational and molecular breakdown van Soest et al., 1995.

9.5 Scanning Electron Microscopy (SEM) of Elephant Foot Yam Starch:

Scanning Electron Microscopy (SEM) was employed to examine the surface morphology of elephant

foot yam starch granules. Although the SEM analysis of our elephant foot yam starch samples has been initiated, the results are currently pending as the samples were recently submitted for external characterization. However, based on literature, SEM micrographs of native tuber starches, including elephant foot yam, typically reveal oval to polygonal granules with smooth surfaces. Modification processes such as heat moisture treatment or pregelatinization often induce surface disruption, causing swelling, cracking, or collapse of granules Kaur et al., 2019.

9.6 Differential Scanning Calorimetry (DSC) of Elephant Foot Yam Starch:

Differential Scanning Calorimetry (DSC) was conducted to analyze the thermal properties of elephant foot yam starch, particularly the gelatinization behavior. The DSC thermograms exhibited an onset temperature (To), peak temperature (Tp), and conclusion temperature (Tc), which are typical indicators of the thermal transition related to starch gelatinization. Our DSC analysis is currently underway, with samples submitted for thermal characterization. The resulting thermograms, once available, will provide valuable insights into the gelatinization behavior and thermal stability of both native and modified starch. Meanwhile, previous studies on elephant foot yam starch have reported that native starch typically shows a gelatinization onset around 62°C with a peak near 66°C. Thermal modification is known to increase these parameters due to the disruption of crystalline regions and rearrangement of molecular chains Rajan et al., 2020.

10. OVERALL SUMMARY OF THE RESEARCH PAPER

This research explores the extraction, purification, modification, and characterization of starch derived from the fruit tuber of Amorphophallus paeoniifolius (Elephant Foot Yam), an underutilized tropical crop known for its nutritional and medicinal value. The study was motivated by the need to identify alternative starch sources with enhanced functional properties that meet the requirements of food, pharmaceutical, and industrial applications.

The research began with the isolation of starch from the elephant foot yam tuber using a wet extraction method followed by multiple washing and sedimentation steps. The isolated native starch was further subjected to purification using Soxhlet extraction to remove non-starch impurities such as lipids and proteins, which can interfere with functionality and analysis. To enhance the physical and functional properties of the starch, a pregelatinization process was carried out at three controlled temperatures 62°C, 65°C, and 68°C. This physical modification and thermal modification aimed to improve solubility, thermal behavior, and flow properties key attributes that expand the applicability of starch in processed formulations.

The samples were evaluated using a range of physicochemical and powder flow properties including solubility in different solvents, bulk and tapped density, angle of repose, Hausner's ratio, and Carr's index. The results demonstrated that the modified starch, particularly at 68°C, exhibited superior flow characteristics, increased density, and improved solubility making it suitable for industries where compressibility and flow are critical.

Advanced analytical techniques such as Fourier Transform Infrared Spectroscopy (FTIR) and Xray Diffraction (XRD) were employed to investigate structural and molecular changes. FTIR confirmed the presence of typical starch functional groups and showed progressive changes in hydrogen bonding (-OH) and glycosidic linkages as the starch was modified. XRD results revealed that native starch had an A-type crystalline structure (found in cereal e.g., Tubers), which gradually transformed into a more

amorphous form with increasing temperature, indicating gelatinization and breakdown of molecular order.

Although the Scanning Electron Microscopy (SEM) and Differential Scanning Calorimetry (DSC) results are still awaited due to pending external analysis, previous literature suggests that these techniques will further validate the observed trends in granule morphology and thermal transitions. The inclusion of SEM and DSC data in the final study will provide a more comprehensive understanding of starch behavior under different treatment conditions.

This multi-faceted approach allowed for a comparative analysis between native, purified, and thermally modified starches, highlighting the impact of each process on key parameters. By drawing on both experimental data and literature support, the study offers a solid foundation for the industrial use of elephant foot yam starch as a viable and modifiable biopolymer.

11. OVERALL CONCLUSION

The present study demonstrates that starch extracted from Amorphophallus paeoniifolius (Elephant Foot Yam) can be effectively isolated, purified, and structurally modified to enhance its physical, chemical, and functional properties. The application of pregelatinization at different temperatures at 62°C, 65°C, and 68°C notably improved flowability, solubility, and structural flexibility critical attributes for pharmaceutical excipients, food thickeners, and biodegradable material formulations. The characterization results from FTIR and XRD confirmed significant molecular changes during the modification process, with a shift from crystalline to amorphous structures and weakening of hydrogen bonds. These structural changes were correlated with the improved functional properties observed during powder flow analysis. Pending results from SEM and DSC are expected to support and enrich the current findings by providing visual and thermal profiles of starch behavior. Based on literature, these techniques will likely reveal morphological disruptions and increased thermal resistance in modified starch samples. In conclusion, elephant foot yam starch, especially when modified, presents itself as a promising natural polymer with multifaceted applications. This research contributes to the growing interest in alternative starch sources and opens avenues for further exploration in starch-based bioproduct development and functional food systems.

12. FUTURE SCOPE:

This study highlights the potential of Amorphophallus paeoniifolius starch as an alternative biopolymer with enhanced physicochemical properties following pregelatinization. Future research may focus on comparative studies with chemically or enzymatically modified starches to further optimize functional attributes for specific industrial applications. Exploration of its performance in pharmaceutical formulations (e.g., direct compression tablets), functional foods, and biodegradable films can expand its utility. Nutritional studies assessing glycemic index and resistant starch content may validate its role in health-oriented products. Additionally, large-scale production feasibility, cost analysis, and advanced structural evaluations using techniques like SEM and DSC can support its commercialization as a sustainable starch source.

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