



(RESEARCH ARTICLE)



Physicochemical Characterization of Unmodified and Modified *Icacina trichantha Oliv* (ITO) Starch

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Abstract

The modification and characterization of starch extracted from *Icacina trichantha Olive* (ITO) tubers, an underutilized plant species indigenous to West Africa was investigated. Native starch was chemically modified via acetylation, and the resultant samples were analyzed using physicochemical tests, Fourier Transform Infrared Spectroscopy (FTIR), Scanning Electron Microscopy (SEM) and Thermogravimetric Analysis (TGA). The acetylated starch exhibited an improved water absorption capacity (2.50 g/g) compared to its unmodified counterpart (2.38 g/g), suggesting enhanced hydrophilicity and swelling potential. Both forms showed similar gelatinization temperatures (78.67°C), indicating consistent thermal resistance. SEM analysis revealed surface disruption and granule roughness post-modification, while FTIR confirmed the successful incorporation of acetyl groups, marked by peak shifts in the fingerprint region. TGA data further highlighted that acetylated starch maintained moderate thermal stability, making it a viable candidate for drilling fluid applications under moderate temperature conditions. Overall, the modifications enhanced key functional properties without compromising the starch's structural integrity, positioning ITO starch as a promising, eco-friendly alternative for industrial applications.

Keywords: Acetylation; *Icacina trichantha Oliv*; Physicochemical property; Starch

1. Introduction

One of the most prevalent carbohydrates in the world is starch, which is a major food source in plants and plant materials. It is the main source of calories and nutritional energy in the majority of foods consumed by humans and serves as the main substrate for human metabolism [1]. The anhydro glucose units that make up this polymeric carbohydrate are mainly bonded by α -D-glycosidic linkages. Amylose (20–30%) and amylopectin (70–80%) make up the majority of the starch granules. In essence, amylose is a linear polymer with α -D-(1-4) glycosidic linkages primarily connecting the anhydro glucose units. Anhydro glucose units connected at α -1,4 and α -1,6 glycosidic linkages (polymers of α -1-4 linked glucopyranose with α -1-6 branches) create the branched amylopectin molecule [2][3]

The chemical composition of starch is affected by the plant's maturity, genetic background, and botanical origin, which can change the amounts of amylopectin and amylose; trace amounts of non-carbohydrate components like proteins, phosphorus, and lipids; and branch chain lengths, all of which contribute to distinct functional characteristics. These variations affect the starch's gelling power, temperature, and viscosity [4]. The chemical structure of starch is composed of linear amylose and highly branched amylopectin, which are present in semi-crystalline granules [5]. Starches can be obtained from fruits, tubers, pseudocereal, grasses, and can be found in the pulps, seeds or culms [6][7]. Starch naturally occurs as discrete particles called granules, with different shapes depending upon the source [1].

The functional and thermal properties of starch, particularly those associated with gelatinization and retrogradation, are among its most crucial features. The process of gelatinization causes the ordered semi-crystalline granules to lose

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their Maltese cross and become entirely amorphous [3]. Because starches are inexpensive, readily available, non-toxic, renewable, biodegradable, and easily convert their physicochemical properties through a variety of alterations and possess ubiquitous hydroxyl groups, they are used in both the food and non-food industries [8]. Because of their structural and functional diversity, starches can be used in a variety of ways [4]. The texture, stability, appearance, and nutritional benefits of different products from food are all greatly impacted by starch [9]. Starch's biological and technical significance is widely recognized, as is its pivotal position in the diet of human. Starches have a vast array of applications in food, such as thickening, stabilizing, texturizing, sticky, Moisture retention, gelling, clouding, dusting, film creation, foam strengthening, anti-staling, and binding [1].

Native undergoes physical, chemical, and enzymatic treatment, which alters its properties because, it has specific restrictions when it comes to its industrial use. This is because the starch's granules readily hydrate, swell, and rupture rapidly, lose their viscosity, and also form cohesive paste. To overcome these restrictions, these starches must be modified, which changes their structure by carefully affecting the hydrogen bonding [7]. Since starch has common hydroxyl groups and simple glycosidic bonds, it can be readily altered physically, chemically, enzymatically, or biotechnologically, or in combination. Its modification is done to introduce specific functionalities to improve film formation, adhesion, texture, gel clarity and sheen, to decrease syneresis, retrogradation, gelling tendencies, to change cooking qualities and improve freeze-thaw stability. Thermal stability, hydrophobicity, amphiphilicity, mechanical strength, and resistance to retrogradation are a few of the characteristics that can be obtained through starch modification [8][4].

Exploring underutilized plant (starch) sources presents a sustainable and economically viable strategy for resource diversification. One such underexplored species is *Icacina trichantha* Oliv (ITO), a perennial tuberous plant native to West and Central Africa. Locally referred to as "urumbia" or "gbegbe" in Nigeria, ITO belongs to the family Icacinaceae and is traditionally cultivated for its starchy tubers, which serve as a food security crop in rural communities [10][11]. The tuber of ITO contains a substantial amount of starch, which has been shown to possess unique physicochemical and functional properties compared to commercial sources such as cassava, potato, and maize. Preliminary studies indicate that its starch exhibits moderate swelling power, high paste clarity, and satisfactory gel-forming ability [12]. These properties make it a potential candidate for industrial applications, including its use as a biodegradable polymer or drilling fluid additive.

The search for biodegradable, renewable, and cost-effective alternatives to synthetic drilling mud additives has led researchers to investigate novel starch sources. Native and modified starches derived from ITO show promise due to their availability, adaptability to modification, and structural compatibility with the functional requirements of drilling fluids. Modified starches, such as carboxymethylated, acetylated, and EDTA-functionalized starches from ITO have demonstrated enhanced gel strength, viscosity control, water loss prevention, and thermal stability, all of which are essential in oil and gas drilling operations.

Notably, carboxymethylated ITO starch has outperformed several conventional additives in maintaining rheological stability under high-temperature conditions, making it particularly suitable for high-pressure, high-temperature (HPHT) well environments [13]. The tuber's starch is also environmentally friendly, decomposing naturally without leaving toxic residues, which aligns with global trends toward greener energy extraction technologies. The research aimed to evaluate the physicochemical properties of acetylated ITO.

2. Materials and methods

2.1. Sample Collection

The tubers of *Icacina Trichantha* Oliv were collected from Abor at Enugu state in the South East Region of Nigeria. All reagents used for the study are of analytical grade.

2.2. Starch Extraction

The tubers of ITO were washed, peeled and cut into smaller sizes and soaked with distilled water for 48 hours. It was grinded with distilled water to paste. The filtrate (suspended starch and distilled water) was received in a bucket, and the paste was diluted with distilled water before being placed into a porous cloth (muslin cloth) and squeezed firmly. The remaining cake was disposed of in an eco-friendly way. After allowing the filtrate to settle for 20 hours, the solid starch collected at the bottom of the bucket and the suspended water was removed. After that, the solid starch was spread out in a pan and was left to dry for 14 days in the sun. After being pounded with a mortar and pestle, the dry starch was subsequently sieved through a 0.424 sieve and placed in a testing container [14].

2.3. Starch Modification (Acetylation)

This was done by dispersing 100 g of the sample in 500 ml of distilled water, and the mixture was continuously stirred for one (1) hour at 30 °C. After that, 3% Sodium Hydroxide was drop-wisely added to the suspension to achieve a pH of 8.0; instantaneously, 12g of acetic anhydride was also added to the mixture at the same time sustaining the pH value within the range of 8.0–8.4. The mixture was stirred continuously for 10 minutes; the pH was adjusted to 4.5 with the addition of 0.5 M Hydrochloric acid. The starch was allowed to settle, and the precipitate formed was rinsed thrice with water, after which 95 % ethanol was used once to neutralize the acid [14].

2.4. Physicochemical characterization

2.4.1. Gelatinization Temperature (°C)

1g of the starch sample was put in a 20 ml beaker and 10 ml of distilled water was added. The dispersion was heated on a hot plate. The gelatinization temperature was then read with a thermometer suspended in the starch slurry [11].

2.4.2. Water Absorption Capacity (%)

The method described by [10] was used to determine the water absorption capacity. 10 ml of water was added to 1g of the starch sample in a centrifuge tube. The mixture was left to stand for 30 minutes, the mixture was centrifuged at 1500 rpm for 20 minutes, and the supernatant was discarded. The tube containing the residue was weighed, and the weight gained was calculated as the water absorption capacity which is expressed as a percentage of the initial weight of the starch.

2.4.3. Determination of pH

In a volumetric flask, 5g of the starch was combined with 20 ml of water, and the mixture was stirred for three minutes. A filter paper was then used to filter the suspension. A pH meter was used to measure the filtrate's acidity and alkalinity.

2.4.4. Thermogravimetric Analysis (TGA)

Thermogravimetric analysis (TGA) using the Shimizu 60-Japan was used to ascertain the samples' thermal stability. In a sample pan with a temperature range of 0–1200 °C and a heating rate of 10 °C/min, 10 mg of starch was added. Sensitivity of +/- 0.14 g, temperature precision of +/- 0.1 °C, mass precision of 0.01%, and low mass furnace accuracy of +/- 0.5%. A nitrogen atmosphere flow rate of was used for the analysis, and the resulting weight loss was noted.

2.4.5. Scanning Electron Microscopy (SEM)

The surface structure of the starch samples was observed by scanning electron microscopy (SEM) analysis and carried out on a scanning electron microscope (SEM/EDS- JOEL - JSM-7600F - USA) using 8000x, 9000x and 10000x magnifications. The starch samples were mounted on aluminum stubs with double sticky tape and coated with platinum coating. The samples were examined and photographed at an accelerating potential of 15 kV.

2.4.6. Fourier Transform Infrared Spectroscopy (FTIR)

The Fourier transform infrared spectroscopy (FTIR) analysis was carried out using FTIR-Nicolet iS10 FT-IR Spectrometer China. The spectral frequency was within the range of 4400 and 350 cm^{-1} with a spectral resolution of 4 cm^{-1} . The IR spectra were analyzed using spectroscopic software Win-IR Pro Version with a peak sensitivity of 2 cm^{-1}

3. Result and discussion

3.1. Physicochemical characterization

The physicochemical properties of ITO starch were evaluated across the unmodified and chemically modified samples specifically acetylated. Key parameters analyzed included pH, gelatinization temperature, and water absorption capacity (WAC), all of which are critical in predicting the behavior of starch. These properties influence the starch's hydration, thermal stability, chemical compatibility, and viscosity development during drilling operations.

Table 1 Physicochemical Properties of ITO

Samples	Physicochemical Properties		
	pH	Gelatinization Temperature	Water Absorption Capacity
Unmodified	5.38	78.67	2.38
Modified	5.79	78.67	2.50

The Acetylated starch (5.79), showed reduction in acidity when compared to the unmodified (5.38), suggesting that acetyl groups ($-\text{COCH}_3$) do not significantly release protons but may slightly reduce the starch's natural acidity by masking hydroxyl sites. The gelatinization temperature indicates the thermal energy required to swell and rupture the starch granules. Acetylated and unmodified starches showed gelatinization temperature (78.67°C), indicating greater internal hydrogen bonding and resistance to swelling. However, while high thermal stability is generally favorable, overly resistant granules may take longer to hydrate during mud preparation. Water absorption is crucial in determining how much liquid the starch can retain, directly affecting its swelling, viscosity, and gel formation. The order observed was: Acetylated (2.50 g/g) > Unmodified (2.38 g/g). Acetylated starch had the highest WAC, suggesting a loose granule structure and greater availability of hydrophilic sites. This favors rapid hydration and swelling, which aligns with its fast-thickening behavior. The unmodified starch had lower but acceptable WAC. Acetylated starch, with the highest WAC and good gelatinization resistance, thickens rapidly but may lack long-term gel stability under pressure.

The physicochemical properties confirmed that chemical modification of ITO starch significantly enhances its functionality. Acetylation, in particular, introduces acetyl group ($-\text{COCH}_3$) groups that optimize gel formation, suspension, and hydration behavior. These traits make it the most suitable candidate for use as a biodegradable, efficient drilling fluid additive, outperforming even conventional carboxymethyl cellulose (CMC) in critical performance metrics.

3.2. Morphology det. Via scanning electron microscopy (SEM)

Scanning Electron Microscopy (SEM) was employed to investigate the surface morphology and elemental composition of native and chemically modified ITO starch samples. The SEM micrographs, alongside Energy Dispersive X-ray Spectroscopy (EDS), revealed the topographical changes and elemental distributions resulting from various modifications. The field of view (FOV), voltage, and detection settings were consistent across samples to ensure accurate comparative analysis.

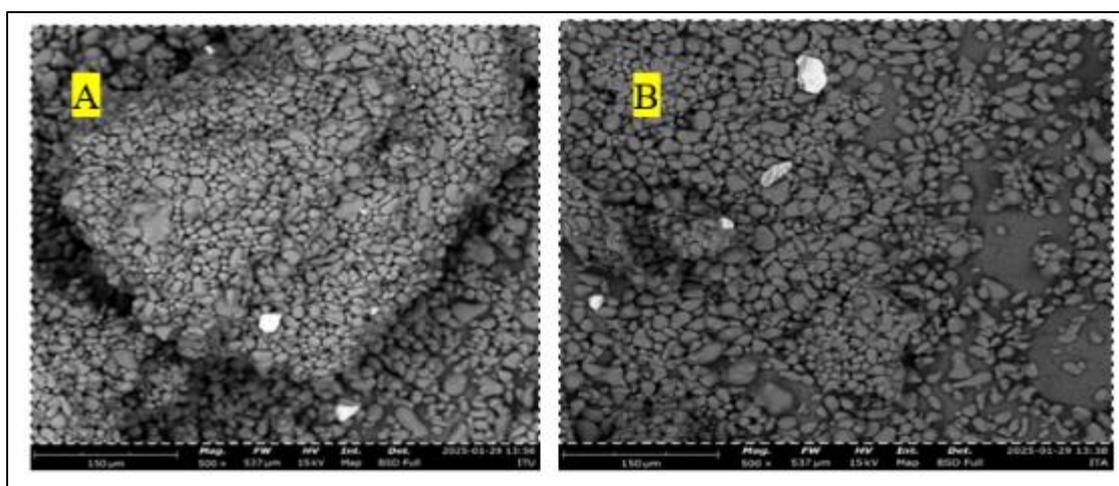
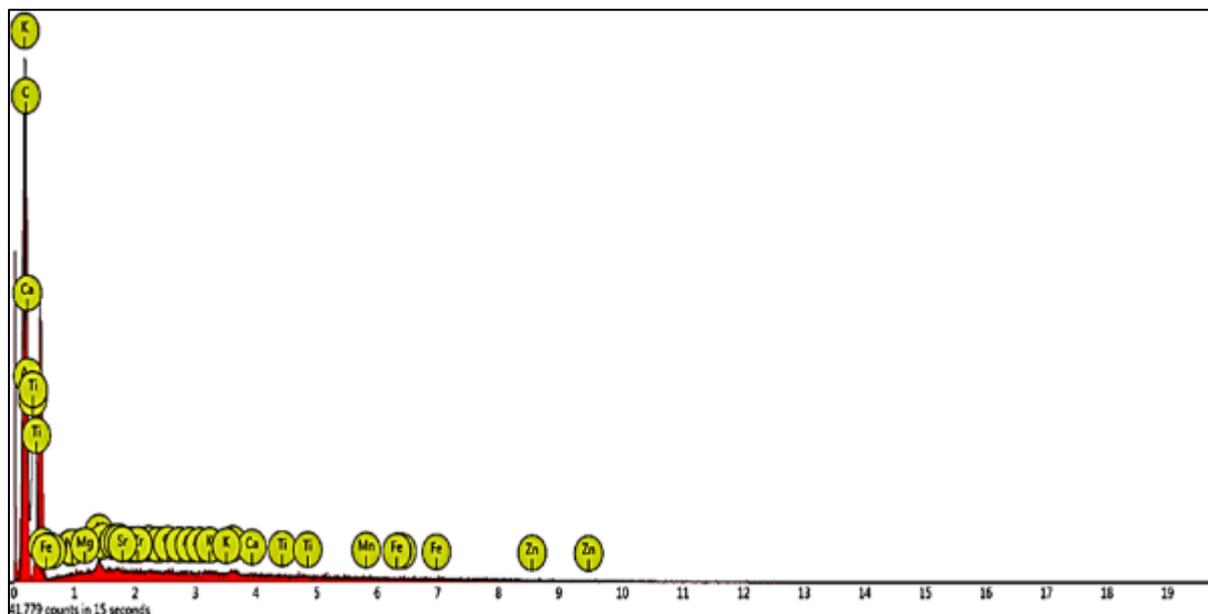


Figure 1 SEM image of (A) unmodified (B) modified ITO starches. FOV: 537 μm , Mode: 15kV - Map, Detector: BSD Ful

Table 2 Compositional analysis of major components in Unmodified starch

Element Number	Element Symbol	Element Name	Atomic Conc.	Weight Conc.
6	C	Carbon	73.45	65.63
7	N	Nitrogen	21.94	22.86
13	Al	Aluminium	2.70	5.43
20	Ca	Calcium	1.00	2.98

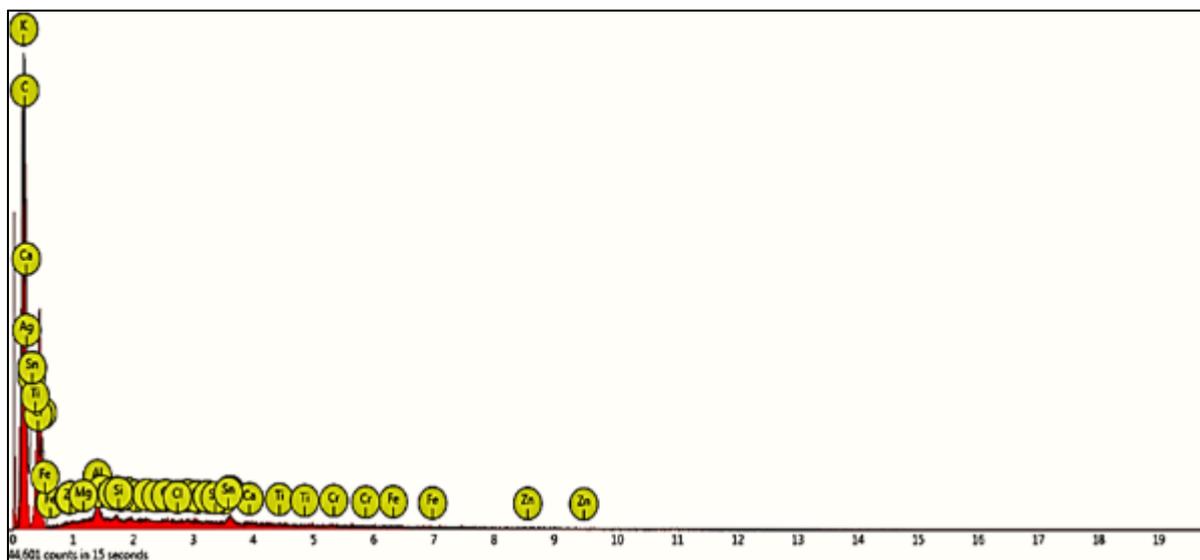
**Figure 2** EDS Spectra of the unmodified starch

The SEM-EDS analysis of the unmodified starch revealed that carbon (C) and nitrogen (N) were the most abundant elements, with atomic concentrations of 73.45% and 21.94%, respectively. Trace elements such as aluminum (Al) (2.70%) and calcium (Ca) (1.00%) were also present. The dominance of carbon is characteristic of polysaccharide-based materials like starch, reflecting the organic nature of the molecule. The nitrogen content may indicate the presence of protein or bound nitrogen-containing compounds. Notably, there was no detectable presence of elements such as sodium (Na), magnesium (Mg), or potassium (K), which are often introduced during chemical modifications. The acetylated sample (ITA) showed the highest atomic percentage of carbon (74.29%) among all samples, likely due to the addition of acetyl groups, which are rich in carbon. Nitrogen content dropped further to 16.15%, which may indicate substitution of nitrogenous moieties or surface masking during acetylation. Elements such as aluminum (3.97%), calcium (2.21%), silver (0.37%), iron (0.59%), and trace amounts of tin (Sn), chromium (Cr), titanium (Ti), and phosphorus (P) were detected. The increased presence of metallic elements suggests interaction with the modifying agents or the formation of surface complexes. Across all samples, carbon and nitrogen were consistently dominant, but the introduction of new elements and changes in elemental concentration after each modification confirmed the successful alteration of the starch granules.

The surface morphology appeared relatively smooth and aggregated, which is typical of native starch granules, and suggests minimal exposure to thermal or chemical stress. The absence of surface deformation supports the granule stability in its unmodified state.

Table 3 Compositional analysis of major components in Modified starch

Element Number	Element Symbol	Element Name	Atomic Conc.	Weight Conc.
6	C	Carbon	74.29	59.04
7	N	Nitrogen	16.15	14.96
13	Al	Aluminium	3.97	7.09
20	Ca	Calcium	2.21	5.85

**Figure 3** EDS spectra of modified starch

SEM imaging of the modified starch revealed a notably smooth small sized with less aggregated or porous surface, consistent with acetylation, which often causes granule swelling or partial gelatinization. These surface changes are desirable in formulations that require enhanced solubility or film-forming properties when compared to the more aggregated unmodified starch. The morphological variations observed from smooth aggregated native granules to increasingly more porous and irregular modified ones, further support the effectiveness of the chemical modifications applied. These changes in surface features and composition are critical as they directly affect the starch's functional properties, such as water absorption, viscosity, binding ability, and suitability for applications in drilling fluid formulations.

3.3. FT-IR analysis

Two samples of *Icacina tricantha Oliv* starch ITU (unmodified), ITA (modified), were analyzed using FTIR in the range 4000–650 cm^{-1} . The observed peaks reflect the molecular features of starch, with a focus on hydroxyl bonding, glycosidic linkages, anhydro-glucose ring vibrations, and skeletal structure

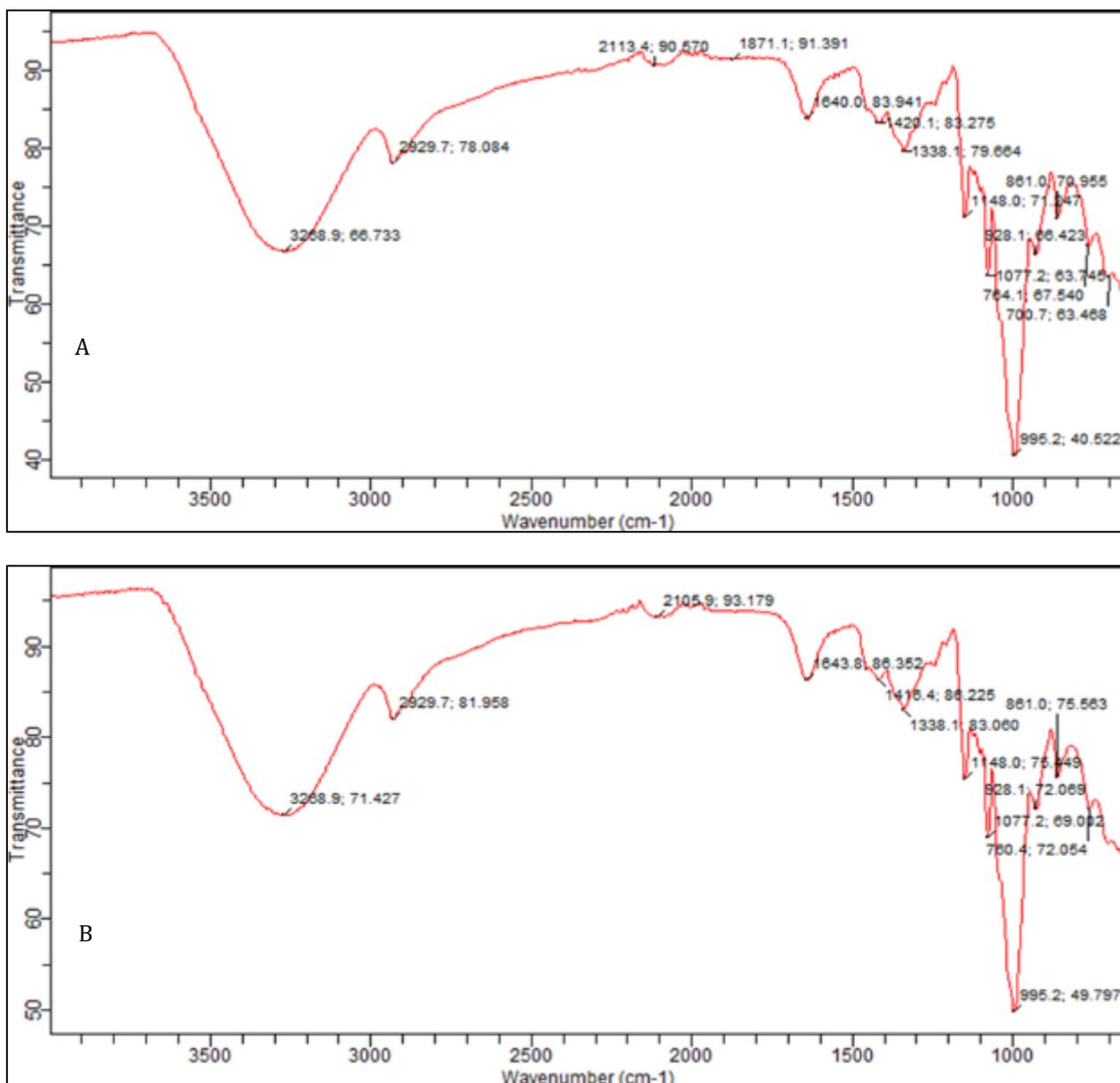


Figure 4 FT-IR Spectra of (a) Modified (b) Unmodified ITO starch

The ITU sample displayed the characteristic fingerprint of native starch. The broad absorption peak at approximately 3415 cm^{-1} is attributed to O–H stretching vibrations, indicating the presence of hydrogen bonding among hydroxyl groups. A smaller peak around 2930 cm^{-1} confirmed C–H stretching from glucose units. The bending vibration of water was seen at 1640 cm^{-1} , showing moisture trapped in the amorphous regions of the starch granules. Three key peaks 1155 cm^{-1} , 1080 cm^{-1} , and 1020 cm^{-1} were observed in the fingerprint region. These peaks are assigned to: C–O–C stretching (glycosidic linkages), C–O stretching in the anhydro-glucose ring, C–O–H bending in amorphous regions, respectively. Further bands below 1000 cm^{-1} , particularly at 930 cm^{-1} , 860 cm^{-1} , and 764 cm^{-1} , reflect skeletal vibrations of the pyran ring and α -(1 \rightarrow 4) glycosidic linkages, typical of native starch.

The FTIR spectrum of ITA retained the major starch peaks observed in ITU but with notable shifts and intensity differences, suggesting molecular reorganization post-modification. The O–H stretch near 3410 cm^{-1} was still present but exhibited a slightly reduced intensity, implying a possible disruption of hydrogen bonds due to physical or chemical treatment.

A mild change in the C–H peak at 2930 cm^{-1} indicated alterations in the hydrophobic character of the starch backbone. In the fingerprint region, the absorbance bands at 1080 cm^{-1} and 1020 cm^{-1} were slightly shifted and broadened, indicating reduced order in the anhydro-glucose rings and an increase in the amorphous content. The skeletal mode at 930 cm^{-1} was less sharp, which may reflect partial hydrolysis or breaking of α -(1 \rightarrow 4) linkages during modification.

3.4. Thermogravimetric analysis (TGA)

Thermogravimetric analysis (TGA) was conducted to evaluate the thermal stability and decomposition behavior of unmodified and chemically modified ITO starches. The thermal resistance of starch is a key indicator of its suitability in high-temperature drilling environments, where additives are expected to maintain structural integrity under circulating heat and downhole pressure. The two starch samples; unmodified (ITU) and acetylated (ITA) were analyzed.

The TGA curves generally showed three primary stages of weight loss across all samples:

- Initial moisture loss (below 150°C): Corresponds to the evaporation of free and bound water molecules.
- Major decomposition (200–350°C): Reflects breakdown of starch macromolecules (amylose and amylopectin), representing the key thermal degradation region
- Final residual breakdown (>350°C): Indicates degradation of carbonaceous residues and trace components.

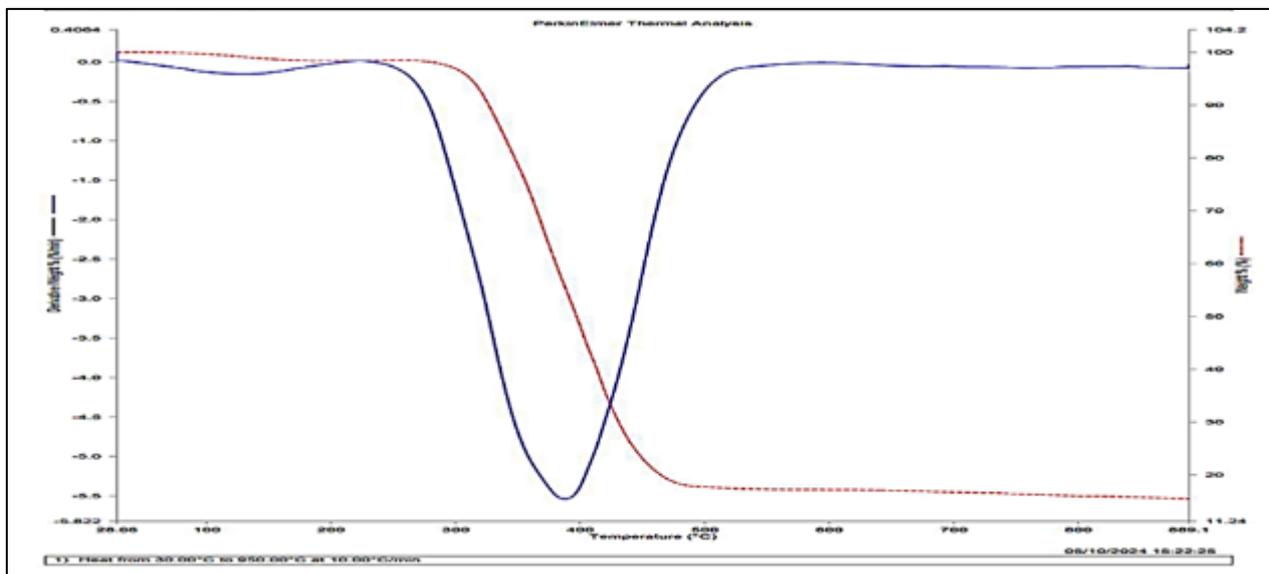
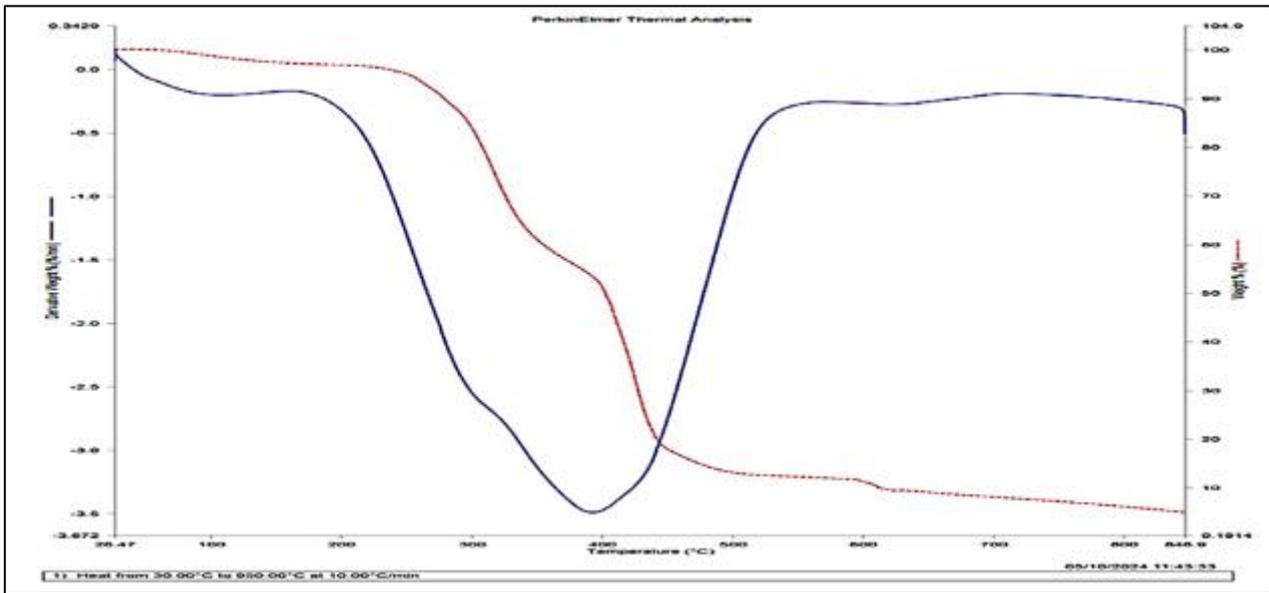


Figure 5 TGA-DTA spectra of the (a) unmodified (b) modified ITO starch

These decomposition stages align with typical starch thermal behavior reported by [13], who noted similar degradation zones in modified polysaccharides used in drilling fluids. Unmodified Starch exhibited a lower onset of decomposition, indicating reduced thermal stability, which suggests that the native granule structure lacks crosslinking or modification to resist heat stress.

Acetylated Starch showed moderate thermal stability, decomposing slightly earlier later than the unmodified sample. Acetylation reduced hydrogen bonding and crystallinity, thereby lowering gelatinization temperature but not significantly enhancing thermal stability. This aligns with its moderate performance in drilling mud tests, offering good flowability but not maximum structural resistance. This modification provides improved swelling and early-stage viscosity, making it suitable for moderate-temperature wells, in agreement with [15] which noted acetylated starch enhances hydration while lowering gelatinization temperature.

Numerous studies have confirmed that modified starch is among the most effective starch modifications for industrial applications. The TGA analysis supports the conclusion that carboxymethylated ITO starch is the most thermally stable and functionally superior candidate for use as a drilling fluid additive. Its delayed degradation onset and sustained weight at higher temperatures make it particularly suitable for deep well or high-temperature drilling operations. Acetylated starch remains a valuable alternative where moderate stability and flow properties are prioritized, while EDTA-modified and native starches are better suited for low-temperature, less demanding environments or cost-sensitive applications.

4. Conclusion

The chemical modification of *Icacina trichantha* Oliv starch through acetylation significantly improved its functional and physicochemical attributes, notably water absorption capacity and hydration behavior, without adversely affecting its gelatinization profile. Analytical techniques confirmed structural reconfigurations and successful acetylation at the molecular level, with surface morphology changes observable via SEM and characteristic shifts in FTIR spectra. Thermogravimetric data revealed moderate thermal resilience, reinforcing its potential for use in moderately demanding industrial environments, such as drilling fluid formulations. These findings underscore the suitability of ITO starch as a biodegradable and cost-effective substitute for conventional additives, contributing to sustainable material sourcing. The promising results also encourage further exploration of ITO in advanced modification routes to tailor its properties for broader commercial applications

Compliance with ethical standards

Disclosure of conflict of interest

The authors declare no conflict of interest to be disclosed.

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