

Investigation of morphological, structural, and thermal changes in palmyra starch (*Borassus flabellifer*) induced by ultrasonication and alcohol-alkali treatment

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ABSTRACT

This study investigates the modification of non-conventional palmyra starch through ultrasonication and alcohol-alkali treatments, assessing their effects on structural, morphological, thermal, and functional properties of native palmyra starch, and comparing them to commercial cassava starch. The results indicate that native palmyra starch shares similar characteristics with cassava starch, including amylose content, granule morphology, crystalline structure, and thermal properties. Modifications significantly altered the whiteness (L^*) value, and amylose content; with ultrasonication achieving the highest amylose content of 32.38 %. A decrease in crystallinity from 25.32 % to 19.12 % was observed, alongside an increase in solubility (11.06–21.56 g/g) and swelling power (14.90–17.69 g/g). The modified palmyra starch exhibited an A-type crystalline structure with diffraction peaks at $2\theta = 15.1^\circ$, 17.1° , 18.0° and 23.0° , which remained uniform with the native starch. FTIR analysis revealed no significant changes in characteristic peaks after ultrasonication and alcohol-alkali treatments. The modifications also resulted in reduced in gelatinization enthalpy (7.98–6.68 J/g), compared to native palmyra starch (12.61 J/g). Overall, the modified palmyra starch demonstrated promising properties, suggesting its potential for a wide range of applications in both food and non-food industries.

1. Introduction

Starch is a highly versatile biopolymer, is widely found in plants, and serves as stable food source for humans. Structurally, starch consists of two main types of glucose polymers: amylose (a linear molecule) and highly branched amylopectin (Reddy et al., 2019). Native starch granules are inherently water-insoluble and exhibit complex semi-crystalline structures, which contribute to their diverse functionalities in food and industrial applications (Liao et al., 2024). The functional properties of starch are affected by different factors, including the amylose-to-amylopectin ratio, granule morphology, crystalline patterns, and the helical structure of amylose (Reddy et al., 2021; Wang et al., 2024). In the food industry, starch plays a crucial role in maintaining the

quality of products during storage (Deng et al., 2020; Xu et al., 2024), while in non-food industries, its cost-effectiveness and renewability make it as functional raw material (Suriya et al., 2018). However, native starches often face limitations such as poor gel strength, high retrogradation, and low paste clarity (Sukhija et al., 2016; Seok et al., 2019). These confronts can be addressed through different modification practices.

Starch modification can be achieved by different practices, including physical, chemical, and enzymatic treatments or a combination of these approaches (Kaul et al., 2023; Sukhija et al., 2016; Suriya et al., 2019). Recently, research into modifying starch to achieve tailored properties has grown significantly. Among modification methods, ultrasonication and alcohol-alkali treatment have gained more interest for modifying

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starches (Kaul et al., 2023; Kunyanee et al., 2023). Ultrasonication a green, and eco-friendly technique, uses acoustic cavitation to physically depolymerize starch, creating cracks and cavities on the starch surface, while also modifying its molecular structure, digestibility, pasting behavior, and rheological properties (Kaul et al., 2023; Yang et al., 2019). In addition to physical treatments, chemical modification techniques, such as alcohol-alkali treatment, are also commonly used to improve starch properties. Alcohol-alkali treatment is valuable as it is a simple and inexpensive method and provides better swelling and solubility at ambient temperature (Kunyanee et al., 2023). In this process, sodium hydroxide disrupts the intermolecular hydrogen bonds within the native starch granules, while ethanol prevents the granules from swelling, preserving their integrity (Akhila et al., 2022). As ethanol evaporates, a cavity is formed in the starch granule and brings about a metastable starch granule with excellent cold water swelling power (Chen et al., 2019).

While research on starch modification has primarily focused on conventional sources such as corn, potato, and rice, there has been limited exploration of starches from underutilized plant sources (Kaul et al., 2023; Sukhija et al., 2016; Suriya et al., 2019). One such potential source is *Borassus flabellifer*, commonly known as palmyra. Widely distributed in tropical regions, particularly in India, palmyra is a multipurpose plant whose young sprouts are rich in starch, dietary fiber, and essential minerals (Krishnaveni et al., 2020). Despite its potential, research on palmyra starch and its modification for industrial and food-related uses remains limited.

The primary aim of this study is to isolate starch from non-conventional palmyra and explore the morphological, structural, and thermal changes induced by ultrasonication and alcohol-alkali treatments. We hypothesize that these modification methods will significantly alter the starch's structural properties, leading to enhanced thermal stability, improved granule morphology, and better functional properties. These modifications are expected to make palmyra starch more suitable for a variety of applications in both food and non-food industries. To test this hypothesis, the structural and functional properties of native and modified palmyra starches will be analyzed using scanning electron microscopy (SEM), X-ray diffraction (XRD), differential scanning calorimetry (DSC), and Fourier-transform infrared spectroscopy (FT-IR). In addition, a comparative analysis with modified cassava starch will be conducted to highlight the advantages and potential uses of palmyra starch.

2. Materials and methods

2.1. Materials

The shoots of palmyra, and tubers of cassava were purchased from the local market in Visakhapatnam, India. Analytical grade chemicals, and reagents were obtained from Hi-Media Co., (Mumbai, India).

2.2. Starch extraction

Starch extraction was performed using an alkaline method, as described by Reddy et al. (2014) with minor modifications. The palmyra and cassava samples (200 g each) were first soaked in 1.0 L of 0.2 % sodium metabisulfite solution and left at room temperature for 10 min. After immersion, the samples were homogenized using a hand blender. The slurry was then sieved through a double-layered cheesecloth and filtered through a 100-mesh sieve. Following, homogenization and filtration, the filtrate was centrifuged at 5000 rpm for 15 min. The sediment was mixed with distilled water in 1:3 (w/v) ratio and subjected to further centrifugation to obtain a white color residue. Finally, the purified starch was dried in hot air oven at 40°C for 24 h. The dried starch were powdered using a motor and pestle and stored at 4°C for further use.

2.3. Proximate composition of starch

The moisture content of the isolated starches was measured using the gravimetric method, based on weight loss after heating at 105°C until constant weight was achieved. The ash, crude fat, and crude protein contents of isolated starches were determined using the muffle furnace method, Soxhlet extraction method and micro-Kjeldahl method, respectively, as described by Reddy et al. (2017).

2.4. Preparation of modified starches

In this study, the ultrasonication and alcohol-alkali treatments were used to prepare modified starches from isolated starches of both palmyra and cassava. The starch modification procedures are detailed below.

2.4.1. Ultrasonication treatment of starch

Ultrasonicated starches were prepared following the method described by Kaul et al. (2023) with minor modifications. Initially, starch (20 % w/v) dispersion was prepared by adding distilled water to the isolated starch. The dispersion was then treated for 30 min using ultrasonic bath (750 W, 20 kHz). In addition, the temperature was maintained at 30°C. After sonication, the suspension was cooled to room temperature, filtered, and dried in an oven for at 45°C for 24 h. The dried starch was powdered, sieved through a 100-mesh sieve, and stored at room temperature for further use. The ultrasonicated starches of cassava and palmyra were labeled as USCAS and USTPS, respectively.

2.4.2. Alcohol-alkali modification of starch

Alcohol-alkali modified starches were prepared following the procedure described by Li et al. (2021) with minor modifications. Initially, a 10 % starch slurry was prepared by adding distilled water to the starch. The slurry was heated in a stirring water bath at 70°C for 5 min. After treatment, the sample was chilled at -20°C for 12 h. The frozen sample was then mixed with 300 mL of anhydrous ethanol and incubated for 2 h in a stirring water bath at room temperature. After incubation, the suspension was centrifuged, and the precipitate was dried in oven for at 45°C for 24 h. The dried starch was powdered, sieved through a 100-mesh sieve, and stored at room temperature for further use. The modified starches of cassava and palmyra by alcohol-alkali treatment were labeled as AMCAS and AMTPS, respectively.

2.5. Amylose content

Amylose content in the starch samples was estimated by the iodometric method, as described by Reddy et al. (2014). The percentage of amylose was calculated according to the following formula (where 3.06 is a conversion factor):

$$\text{Amylose}(\%) = \text{Absorbance} * 3.06 * 20$$

2.6. Morphological characteristics

The morphological characteristics of starch granules was observed using scanning electron microscopy (SEM, Tescan Mira 3, Czech Republic) at an accelerating voltage of 5 kV. Prior to analysis, each sample was evenly distributed on a sample stub and coated with gold under vacuum by using a sputter coater. Observations were carried out under a high-vacuum environment at a magnification of 1500^X.

2.7. X-ray diffraction (XRD)

The crystalline patterns of each starch sample were analyzed using an X-ray diffractometer (Bruker D8 Advance, Germany) with a Cu K α source and a wavelength of $\lambda = 1.5406 \text{ \AA}$ operating at 40 kV and 40 mA. The diffraction angle (2θ) was scanned from 10 to 50° at a scanning rate of 1.5°/min. The relative crystallinity of starches was calculated using

the following formula:

$$\text{Relative crystallinity}(\%) = \frac{\text{Crystalline area}}{\text{Amorphous area} + \text{Crystalline area}} * 100$$

2.8. Color analysis

Hunter-Lab Spectrophotometer (Hunter D-25, Reston, Virginia, USA) was used to measure the color parameters of the samples. Approximately 5.0 g of sample was placed in a glass container positioned over the slit of the instrument. The color indicators, L^* (lightness), a^* (redness) and b^* (yellowness) of native and modified starches were recorded.

2.9. Fourier transform infrared (FTIR) analysis

FTIR spectra of the starch samples were obtained using an FTIR spectrometer (ALPHA II, Bruker, Germany) in the range of 500–4000 cm⁻¹ at room temperature. The potassium bromide disk method was used to prepare the samples for FTIR analysis.

2.10. Water activity

An Aqualab 4 TE (METER Group, Pullman, USA) was used to measure the water activities of each starch sample, and the analysis was performed at room temperature.

2.11. Functional properties

The functional properties, including swelling power (SP) and solubility, of each starch sample were analyzed following the method described by Núñez-Bretón et al. (2024). A 10 mg/mL starch suspension was prepared by adding distilled water to the starch. The dispersion was then heated at 90°C in a water bath for 30 min with constant stirring. After incubation, the samples were cooled and centrifuged at 5000 rpm for 15 min. The supernatant was dried in an oven at 105°C for 2 h, and the wet sediment was used to calculate the swelling power (SP) and solubility using the following equations:

$$\text{Solubility}(\%) = \frac{\text{Solids in supernatant(g)}}{\text{Sample weight(g)}} * 100$$

$$\text{SP} = \frac{\text{Gel weight(g)}}{\text{Sample weight(g)} - \text{Solids dissolved in supernatant(g)}}$$

2.12. Differential scanning calorimetry (DSC)

Thermal transition characteristics of the starch samples were analyzed using a differential scanning calorimeter (DSC-60 Plus, SHIMADZU, Japan). Approximately 5.0 mg of sample was placed in an aluminum pan, and the pan was heated from 30°C to 200°C at a rate of 10 °C/min. The gelatinization temperature of To (onset), Tp (peak), Tc (conclusion), and enthalpy change (ΔH) were recorded.

2.13. Statistical analysis

Data were analyzed using SPSS software (IBM, Inc., New York, NY). The results from triplicate analyses were subjected to analysis of variance (ANOVA) followed by the Duncan's Multiple Range Test (DMRT) to determine statistical significance ($p < 0.05$) among the different treatments. The correlation analysis between starch properties was determined by SPSS software.

3. Results and discussion

3.1. Chemical composition of native starches

The results of the chemical composition of native starches isolated from palmyra and cassava are mentioned in Table 1. The moisture content of the isolated starches shown similar values was 10.64 % (palmyra) and 10.56 % (cassava), which is uniform with the moisture content of starches isolated from other tubers (Lin et al., 2020; Suriya et al., 2019). Also, the low levels of non-starch components including protein, fat and ash contents in isolated starches may reflect the efficiency of isolation process for purifying the starch. These results indicate that the starches isolated from palmyra, and cassava were of high purity and suitable for further modification and use in food and industrial applications.

3.2. Amylose content

The amylose content of both native and modified starches is presented in Table 2. It is well-established that higher amylose content in starch leads to stronger gelling and high thickening power, making it ideal for various starch-based products (Wang et al., 2024). In this study, the results indicate that modification practices, including ultrasonication and alcohol-alkali treatment improved the levels of amylose in native starches. The amylose content of palmyra starch increased significantly from 23.22 % to 25.69 % after ultrasonication, with a similar increase observed in cassava starch (21.59–28.47 %). This increase in amylose content is credited to amylose leaching during ultrasonication (Kaul et al., 2023), as well as partial depolymerization of both amylose and amylopectin, which may promote the formation of more linear chains, thereby increasing amylose content (Kunyanee et al., 2023). Further, after alcohol-alkali treatment, the amylose content of palmyra starch increased significantly from 23.22 % to 32.38 %, with a similar increase seen in cassava starch (21.59–28.02 %). These changes might be due to the degradation of the crystalline regions of the starch granules during alkali treatment, which facilitated the release of amylose (Akhila et al., 2022).

3.3. Morphological characteristics

The morphology of native and modified starch granules, as treated by ultrasonication and alcohol-alkali treatments, is shown in Fig. 1. The native palmyra starch (NTPS) granules (Fig. 1A) were predominantly elliptical and irregular in shape, with a smooth surface and no apparent structural damage. Similarly, the native cassava starch (NCAS) granules (Fig. 1D) exhibited an oval to irregular shape, featuring prominent surface dentations (Lin et al., 2020). After ultrasonication, there were no significant changes in the size and shape of the starch granules from either cassava or palmyra. SEM images of ultrasonicated palmyra starch (USTPS, Fig. 1B) and ultrasonicated cassava starch (USCAS, Fig. 1E) showed that while their structural integrity was largely maintained, some granules exhibited small fissures and depressions. The results were found to be in line with a study carried out by Kaul et al. (2023) and Ramos et al. (2024). Additionally, alcohol-alkali treatment caused

Table 1
Proximate composition of isolated starches from cassava and palmyra.*

Parameters**	Proximate composition***			
	Moisture (%)	Ash (%)	Fat (%)	Protein (%)
NTPS	10.64 ± 0.92 ^a	0.13 ± 0.01 ^b	0.06 ± 0.01 ^a	0.45 ± 0.03 ^a
NCAS	10.56 ± 0.64 ^a	0.17 ± 0.01 ^a	0.05 ± 0.02 ^a	0.38 ± 0.05 ^a

*NTPS, and NCAS indicate native palmyra starch, and native cassava starch, respectively.

** Values expressed mean ± standard deviation. Data of different alphabets in the same column were different with statistically significant ($p < 0.05$).

Table 2

Amylose content, relative crystallinity, and color of native and modified starches.*

Parameter**	Type of starches***					
	NTPS	USTPS	AMTPS	NCAS	USCAS	AMCAS
Amylose (%)	23.22 ± 0.67 ^d	25.69 ± 0.02 ^c	32.38 ± 0.75 ^a	21.59 ± 0.80 ^e	28.47 ± 0.69 ^b	28.02 ± 8.23 ^b
RC (%)	25.32 ± 0.35 ^b	24.73 ± 0.36 ^b	19.12 ± 0.14 ^d	27.70 ± 0.81 ^a	25.36 ± 0.39 ^b	22.82 ± 0.97 ^c
L*	94.99 ± 0.64 ^b	96.96 ± 0.62 ^a	93.89 ± 0.61 ⁱ	96.47 ± 0.34 ^a	96.93 ± 0.47 ^a	96.47 ± 0.05 ^a
a*	-0.13 ± 0.02 ^d	-0.12 ± 0.01 ^d	-0.06 ± 0.03 ^c	0.18 ± 0.01 ^a	0.08 ± 0.03 ^b	0.08 ± 0.02 ^b
b*	1.33 ± 0.18 ^b	1.17 ± 0.05 ^c	1.27 ± 0.09	2.13 ± 0.01 ^a	2.06 ± 0.03 ^a	2.08 ± 0.02 ^a

**RC indicate relative crystallinity.

***NTPS, USTPS, AMTPS, NCAS, USCAS, and AMCAS indicate native palmyra starch, Ultrasonicated palmyra starch, alcohol-alkali treated palmyra starch, native cassava starch, ultrasonicated cassava starch, and alcohol-alkali treated cassava starch, respectively.

* Values expressed mean ± standard deviation. Data of different alphabets in the same column were different with statistically significant ($p < 0.05$).

noticeable changes and irregularities in the starch granules, likely due to shrinkage following swelling during the treatment (Akhila et al., 2022).

3.4. X-ray diffraction

Fig. 2 shows the diffractograms of native and modified starches. Native palmyra starch (NTPS) showed a single peak at an angle (2θ) of 15.1° , and 23.0° , and a doublet (2θ) at 17.1° , and 18.0° , indicating an A-type starch pattern, and a similar pattern was witnessed for NCAS. After ultrasonication and alcohol-alkali treatments, the crystalline patterns of both palmyra (Fig. 2A) and cassava (Fig. 2B) starches persisted mostly unchanged, indicating that these modification treatments had least

effect on the crystalline structure of the starch granules. These findings align with the study of Ramos et al. (2024), who reported that similar treatments did not lead to significant alterations in the crystalline portion of starch granules. The continuity of the A-type pattern implies that these treatments did not disrupt the basic crystalline architecture of the starch granules. The relative crystallinity (RC) of both native and modified starches is presented in Table 2. The RC of NTPS and NCAS shown significant difference and the values are 25.32 % (NTPS) and 27.70 % (NCAS), respectively. After modification treatment, a minor decrease was observed in ultrasonicated starches (Table 2), indicating that the ultrasonication may have damaged the crystalline portion of the starch granules (Kaul et al., 2023). Interestingly, a major change in crystallinity was observed in alcohol-alkali treated starches where the crystallinity reduced significantly (Table 2). Similar results were reported by Choi et al. (2017), who suggested that alcohol-alkali treatment led to decrease in crystalline portion and resulted in more amorphous granular starch formation.

3.5. Color analysis

The visual attributes of the product can be determined by color parameters and play a significant role in their commercial appeal (Suriya et al., 2019). Table 2 shows a significant ($p < 0.05$) change in all types of starch with respect to color parameters. All the starch samples, both native and modified, showed high L^* values (>94), inferring the samples were whiter in form. After ultrasonication, the L^* values of USTPS (96.96) and USCAS (96.93) increased significantly. This increase in L^* was accompanied by a decrease in b^* , indicating that ultrasonication reduced the yellowness of starch granules. A similar trend was observed for the starches that underwent alcohol-alkali treatments (Kaul et al., 2023). These modified starches also showed a rise in L^* values, suggesting an increase in L^* , and a decrease in both a^* and b^* values. The decrease in a^* and b^* values following alcohol-alkali treatment could be attributed to the breakdown or separation of pigments and other colored compounds that may have been present in the native starches (Akhila

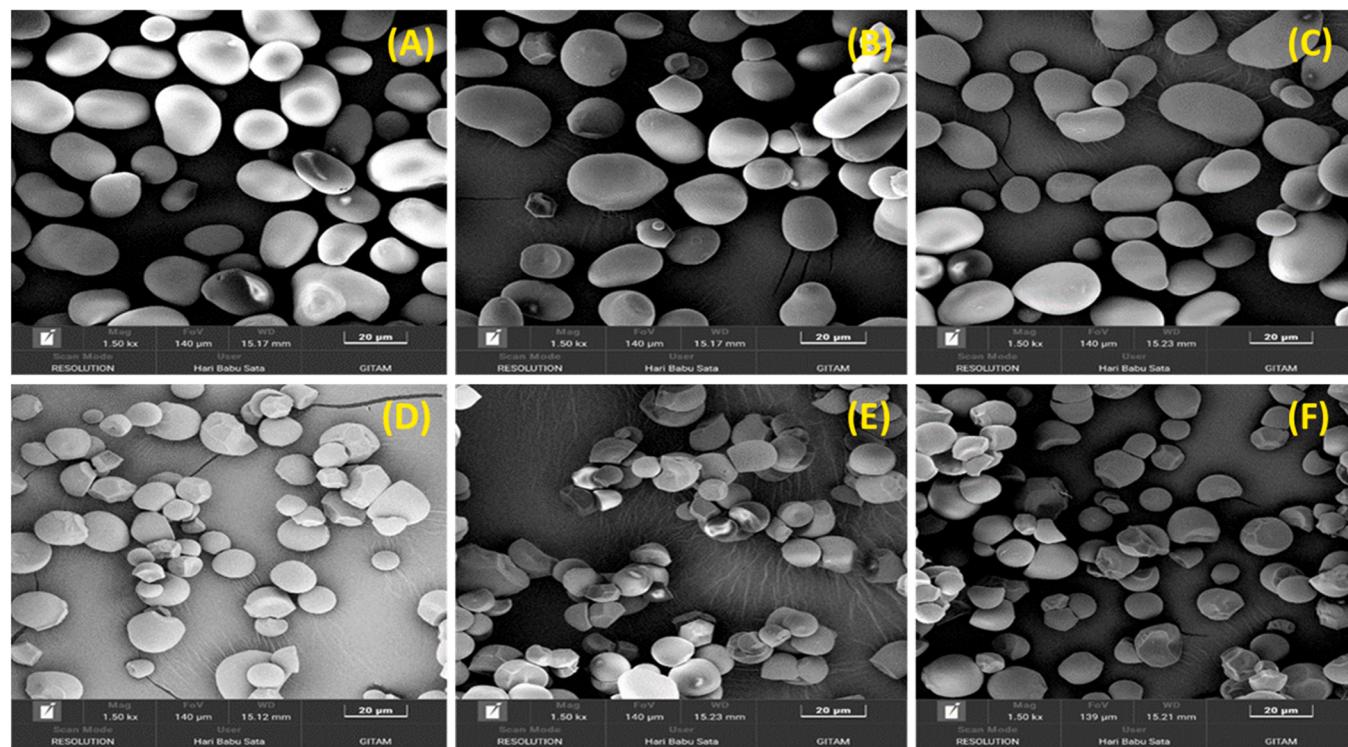


Fig. 1. FE-SEM photographs of native and modified starches of palmyra and cassava. (A) native palmyra starch; (B) Ultrasonicated palmyra starch; (C) alcohol-alkali treated palmyra starch; (D) native cassava starch (E) ultrasonicated cassava starch and (F) alcohol-alkali treated cassava starch, respectively.

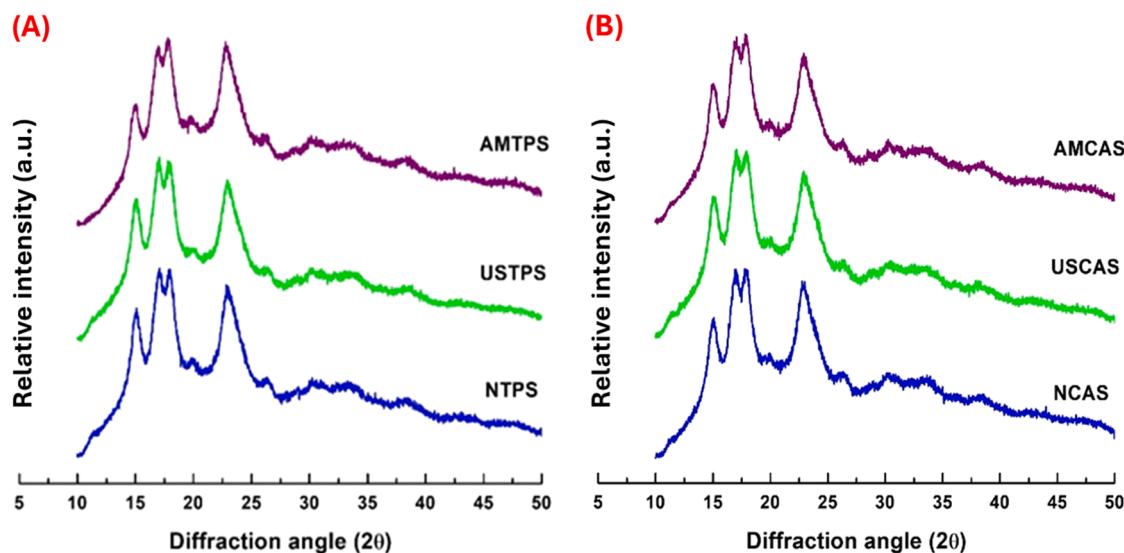


Fig. 2. XRD patterns of native and modified starches of tender palmyra shoot (A) and cassava (B). NTPS, USTPS, AMTPS, NCAS, USCAS, and AMCAS indicate native palmyra starch, Ultrasonicated palmyra starch, alcohol-alkali treated palmyra starch, native cassava starch, ultrasonicated cassava starch, and alcohol-alkali treated cassava starch, respectively.

et al., 2022). Furthermore, these results suggest that the modification treatments not only affect the structure of the starch but also enhance its visual qualities, which could be beneficial for the commercialization of starch-based products.

3.6. Fourier transform infrared (FTIR) spectroscopy analysis

The FT-IR spectra of native and modified starches were analyzed to explore the structural changes induced by ultrasonication and alcohol-alkali treatments. The spectra of both native and modified starches exhibited similar absorption peaks, but notable changes in the amplitude of these peaks were observed, reflecting changes in the starch structure (Fig. 3). For the native starches (cassava and palmyra), several characteristic absorption bands were identified. A prominent band around 1648 cm^{-1} was attributed to the bound water within the starch molecule, which is typical of starch's hydroscopic nature (Suriya et al., 2019). Also, a broad peak at 3285 cm^{-1} was observed, corresponding to the hydrogen-bonded hydroxyl groups, which are present in the

hydroxyl groups of the starch chains (Reddy et al., 2021). The sharp peak around 995 cm^{-1} was assigned to the vibrational stretching C–O bonds (Kaul et al., 2023), and the deeper band at around 990 cm^{-1} was linked to the α -1, 4 glycosidic linkages and C–O–C bond vibrations, which are indicative of the amylose and amylopectin structure in starch (Babu et al., 2019).

After undergoing ultrasonication and alcohol-alkali treatments, the modified cassava and palmyra starches exhibited FT-IR spectra that were quite similar to their native counterparts, although with differences in the amplitudes of the absorption bands. Specially, the absorption band for the hydroxyl group stretching shifted slightly to around 3300 cm^{-1} , and this was accompanied by a broad peak, suggesting an increased formation of hydrogen bonds in modified starches (Suriya et al., 2018). The shift and broadening of this peak indicate a structural modification, which likely reflects the reorganization of the starch microstructure during the modification treatments (Akhil et al., 2022). The reduction in the amplitude of the absorption bands observed after both ultrasonication and alcohol-alkali treatments further suggests that

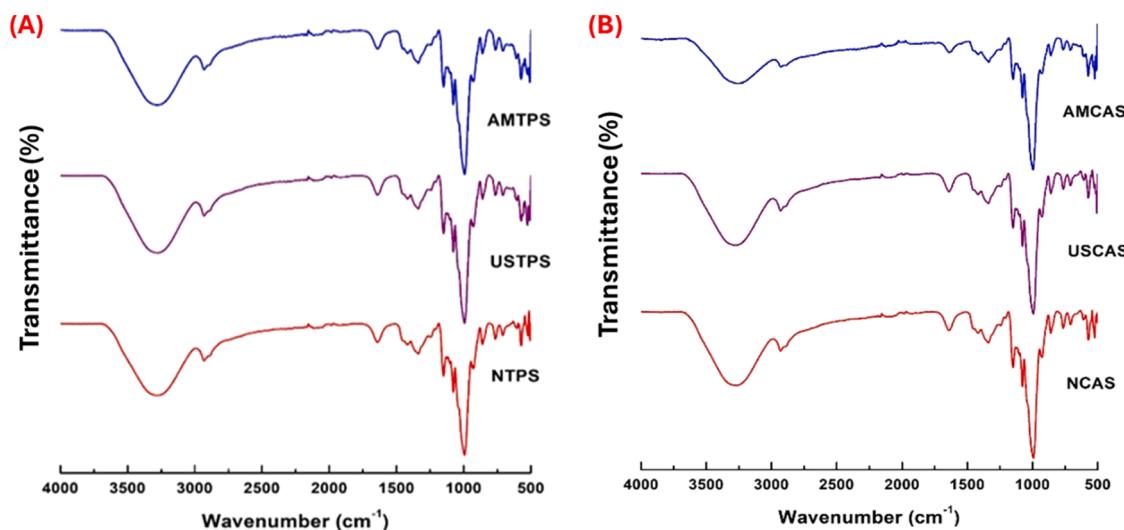


Fig. 3. FT-IR spectra of native and modified starches of tender palmyra shoot (A) and cassava (B). NTPS, USTPS, AMTPS, NCAS, USCAS, and AMCAS indicate native palmyra starch, Ultrasonicated palmyra starch, alcohol-alkali treated palmyra starch, native cassava starch, ultrasonicated cassava starch, and alcohol-alkali treated cassava starch, respectively.

changes in the structural configuration of the starch molecules occurred during these treatments (Kaul et al., 2023). The modifications likely caused a disruption or reordering of the starch chains, leading to a decrease in the intensity of certain vibrational bands (Cao and Gao, 2020). This reduction in intensity could be due to a decrease in hydrogen bonding and structural rearrangement in the starch, potentially attributed to the breaking of intermolecular interactions and the realignment of the starch macromolecules during treatment (Kaul et al., 2023).

3.7. Water activity

Water activity is a key factor influencing the stability of starch-based products. It plays an important role in determining aspects such as lipid oxidation, enzyme activity, and microbial growth in foods (Charles et al., 2021). The water activity values of both native and modified starches are shown in Table 3. Native and modified palmyra starches exhibited water activity values ranging from 0.43 to 0.52, while for cassava starches ranged from 0.49 to 0.55. The differences in water activity for native starches may be attributed to variations in starch variety (Veiga-Santos et al., 2005). Also, the alcohol-alkali treated starches (AMTPS and AMCAS) exhibited lower water activity values compared to the ultrasonicated starches (USTPS and USCAS). This variance might be due to the difference in parameters of starch modification treatments. Overall, the variance in water activity values suggest that the modified starches, particularly those treated by alcohol-alkali method, may offer better storage stability compared to the ultrasonicated starches.

3.8. Functional properties

The swelling power of both native and modified starches is presented in Table 3. Following ultrasonication, the swelling power for palmyra starch increased from 14.90 to 15.40 g/g, and for cassava starch it increased from 12.44 to 15.76 g/g. The increase in swelling power was attributed to the structural changes in the starch by ultrasonication, easing the movement of water molecules and disrupting the free hydroxyl groups of amylose (Kaul et al., 2023). Moreover, the release of amylose molecules during ultrasonication may further contribute to the increase in volume, as amylose tends to absorb more water compared to amylopectin (Kumar et al., 2014). Alcohol-alkali treatment showed a significant increase in swelling power of native starches. For palmyra starch, the swelling power increased markedly from 14.90 g/g to 17.69 g/g, and a similar trend was observed for cassava starch (12.44–16.65 g/g). After alcohol-alkali treatment, the increase in swelling power might be due to the increased surface area and porosity of the starch granules, and which enhance the interaction between the starch and the surrounding water, further improving its swelling power (Chen et al., 2019). These modifications can have important implications for starch-based products in food and industrial applications.

From the results presented in Table 2, the solubility values of the starch granules increased significantly from 9.97 to 19.36 g/g for cassava, and from 11.06 to 21.56 g/g for palmyra, respectively. This increase can be attributed to break down the starch structure by

ultrasonication, enhancing the leaching and movement of amylose chains, which results in higher solubility (Yang et al., 2019). Alcohol-alkali treatment also resulted in an increase in solubility, though to a lesser extent than ultrasonication. For cassava starch, solubility increased from 9.97 to 17.33 g/g, and for palmyra starch, it amplified from 11.06 to 17.23 g/g. This change might be due to the weakening of hydrogen bonds within the starch granules and potential degradation of amylopectin (Chen et al., 2019), which exposes more hydroxyl groups and allows the starch to swell and disperse more readily in water (Kaul et al., 2023). The alcohol-alkali treatment likely led to the disruption of amylopectin's crystalline structure, enhancing the starch's ability to dissolve in water (Chen et al., 2019).

3.9. Thermal properties

The thermal properties of native and modified starches are shown in Table 4 and Fig. 4. Results revealed that the NTPS had higher gelatinization temperatures (T_g , T_p , and T_c) than NCAS granules. The difference in thermal parameters of native starch granules could be possible due to the difference in starch structure, and the ratio of amylose and amylopectin (Deng et al., 2020). After ultrasonication and alcohol-alkali treatments, the gelatinization temperatures of both modified starches significantly reduced when compared to native starches (Table 4). After modification, the reduction in thermal parameters and the enthalpy (ΔH) change values of starch granules could be possible due to the starch structural changes during ultrasonication and alcohol-alkali treatments (Kaul et al., 2023), which further decreases the porosity of the channels in the granules after the treatment. Further, the destruction of starch by modification treatment enhanced the movement of water molecules into starch granules and raised the irreversible hydration of the starch granule (Hu et al., 2019).

3.10. Correlation analysis among different physicochemical properties

Correlation analysis revealed that amylose content was not correlated with gelatinization temperature and enthalpy, but it was positively correlated with swelling power ($r = 0.73$, $p < 0.001$) and solubility ($r = 0.57$, $p < 0.05$) and negatively correlated with other parameters including water activity and crystallinity. From the findings (Table 5), swelling power was positively correlated with solubility ($r = 0.48$, $p < 0.05$) and negatively correlated with crystalline parameter. Relative crystallinity had no significant correlations with gelatinization temperature and enthalpy (Table 5), and it was positively correlated with water activity ($r = 0.72$, $p < 0.001$).

4. Conclusion

In this study, non-conventional palmyra starch was successfully modified using ultrasonication and alcohol-alkali treatments, and the effects on its structural, thermal, and functional properties were thoroughly evaluated and compared with commercial cassava starch. The results demonstrated that while native palmyra starch shares several characteristics with cassava starch, such as amylose content, granule

Table 3
Water activity and functional properties of native and modified starches.*

Parameter**	Type of starches***					
	NTPS	USTPS	AMTPS	NCAS	USCAS	AMCAS
Water activity	0.50 ± 0.01 ^c	0.52 ± 0.01 ^b	0.43 ± 0.02 ^e	0.52 ± 0.01 ^{bc}	0.55 ± 0.01 ^a	0.49 ± 0.01 ^d
SP (g/g)	14.90 ± 1.17 ^{ab}	15.40 ± 2.81 ^a	17.69 ± 1.23 ^a	12.44 ± 0.86 ^{bc}	15.76 ± 0.67 ^a	16.65 ± 0.96 ^a
Solubility (g/g)	11.06 ± 2.03 ^c	21.56 ± 1.18 ^a	17.23 ± 3.07 ^b	9.97 ± 0.82 ^c	19.36 ± 1.56 ^{ab}	17.33 ± 0.87 ^b

*SP indicate Swelling power.

**NTPS, USTPS, AMTPS, NCAS, USCAS, and AMCAS indicate native palmyra starch, Ultrasonicated palmyra starch, alcohol-alkali treated palmyra starch, native cassava starch, ultrasonicated cassava starch, and alcohol-alkali treated cassava starch, respectively.

* Values expressed mean ± standard deviation. Data of different alphabets in the same column were different with statistically significant ($p < 0.05$).

Table 4

Thermal properties of native and modified starches.*

Parameter**	Type of starches***					
	NTPS	USTPS	AMTPS	NCAS	USCAS	AMCAS
T _O (°C)	76.62 ± 0.67 ^a	69.07 ± 2.03 ^b	74.04 ± 0.83 ^a	74.18 ± 2.93 ^a	67.92 ± 1.52 ^b	70.52 ± 1.16 ^b
T _p (°C)	94.89 ± 0.73 ^a	85.02 ± 1.02 ^d	90.41 ± 0.38 ^b	88.19 ± 1.20 ^c	86.36 ± 1.04 ^d	78.42 ± 0.51 ^e
T _c (°C)	107.21 ± 0.66 ^a	101.53 ± 0.49 ^{cd}	105.55 ± 0.61 ^b	100.49 ± 0.49 ^d	102.49 ± 0.50 ^c	95.18 ± 0.94 ^e
ΔH (J/g)	12.61 ± 0.95 ^a	6.68 ± 1.10 ^b	7.94 ± 1.03 ^b	13.48 ± 0.71 ^a	7.69 ± 0.30 ^b	5.42 ± 0.54 ^{bc}

* * T_O, T_p, T_c, and ΔH indicate onset, peak, conclusion temperatures, and enthalpy, respectively.

** NTPS, USTPS, AMTPS, NCAS, USCAS, and AMCAS indicate native palmyra starch, Ultrasonicated palmyra starch, alcohol-alkali treated palmyra starch, native cassava starch, ultrasonicated cassava starch, and alcohol-alkali treated cassava starch, respectively.

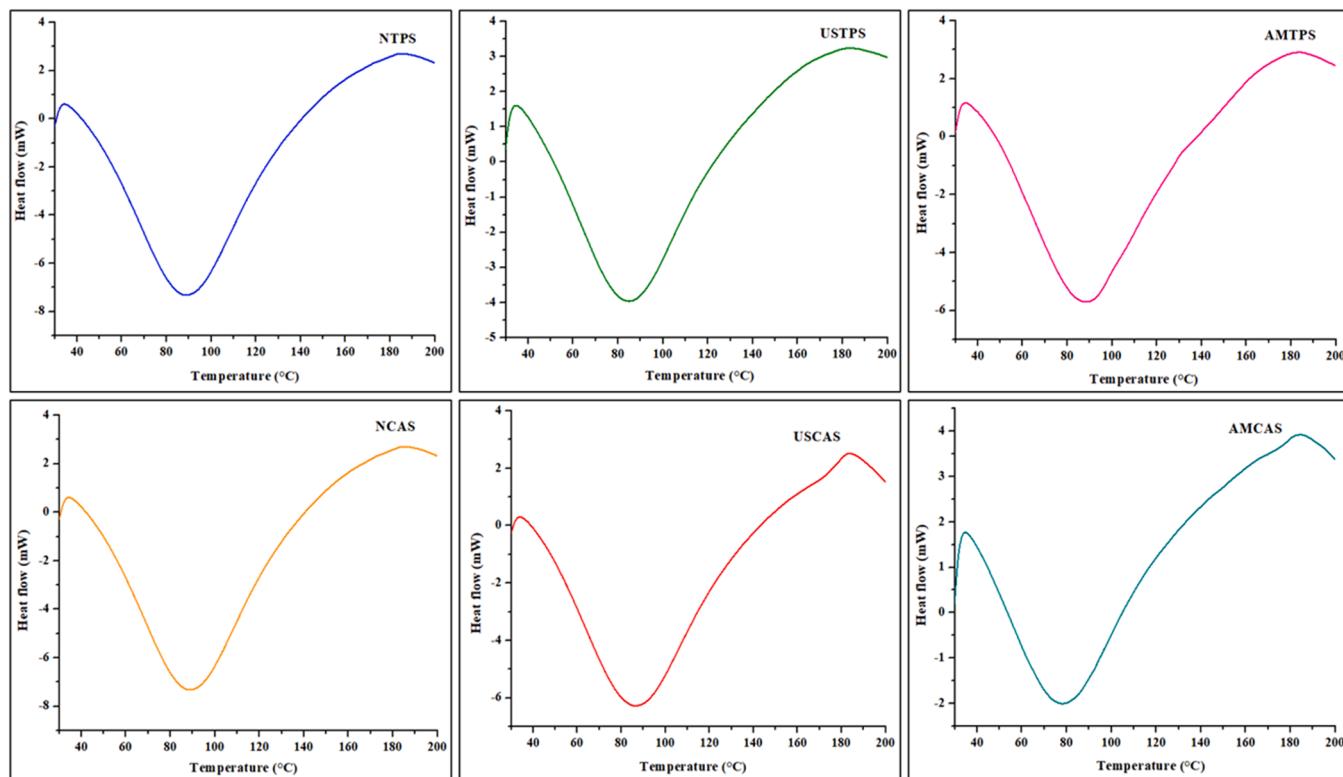
* Values expressed mean ± standard deviation. Data of different alphabets in the same column were different with statistically significant ($p < 0.05$).

Fig. 4. DSC curves of native and modified starches of cassava and palmyra. NTPS, USTPS, AMTPS, NCAS, USCAS, and AMCAS indicate native palmyra starch, Ultrasonicated palmyra starch, alcohol-alkali treated palmyra starch, native cassava starch, ultrasonicated cassava starch, and alcohol-alkali treated cassava starch, respectively.

Table 5

Pearson correlation coefficients among different physicochemical and functional properties of native and modified starches.

Parameter***	SP	WA	S	Amylose	RC	T _O	T _p	T _c	ΔH
SP	1								
WA	-0.46	1							
S	0.48*	0.04	1						
Amylose	0.73**	-0.56*	0.57*	1					
RC	-0.50*	0.72**	-0.29	-0.71**	1				
T _O	-0.22	-0.41	-0.71**	-0.33	-0.16	1			
T _p	-0.18	-0.13	-0.50*	-0.22	0.02	0.66**	1		
T _c	0.05	-0.16	-0.21	0.26	-0.07	0.46	0.93**	1	
ΔH	-0.25	-0.17	-0.03	-0.07	0.02	-0.13	-0.54*	-0.66**	1

*** SP, WA, S, RC, T_O, T_p, T_c, and ΔH indicate swelling power, water activity, solubility, relative crystallinity, onset, peak, conclusion temperatures, and enthalpy, respectively.*, and ** means the correlations are significant at $P < 0.05$, and $P < 0.01$ levels, respectively.

morphology, and crystalline structure, the treatments applied significantly altered its properties. Notably, the ultrasonication and alcohol-alkali treatments resulted in increased solubility and swelling power,

reduced crystallinity, and altered gelatinization behavior, which collectively enhanced the starch's functional properties. These changes suggest that palmyra starch, with its modified characteristics, could

offer new opportunities for diverse applications in both food and non-food industries. Future studies are needed to further investigate the mechanisms underlying these changes and to explore the long-term stability and performance of modified palmyra starch in various practical applications. Overall, the findings of this study support the potential of non-conventional palmyra starch as a viable alternative to commercial starches for a variety of industrial uses.

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CRediT authorship contribution statement

Tamma Medha: Writing – original draft, Visualization, Validation, Software, Methodology, Investigation, Formal analysis, Data curation, Conceptualization. **Chagam Koteswara Reddy:** Writing – review & editing, Supervision, Resources, Project administration, Methodology, Investigation, Funding acquisition, Formal analysis, Conceptualization. **Hemasundar Alavilli:** Writing – review & editing, Validation. **Challa Surekha:** Writing – review & editing, Visualization, Validation. **Tapasya Kumari:** Writing – review & editing, Validation, Software. **Muhammad Nazim:** Funding acquisition, Formal analysis, Writing – review & editing. **Hosam O. Elansary:** Funding acquisition, Formal analysis, Writing – review & editing.

Declaration of Competing Interest

The authors declare that they have no conflict of interest.

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Data availability

Data will be made available on request.

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