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Modified Banana Starch: A Promising Pharmaceutical Disintegrant for the Future

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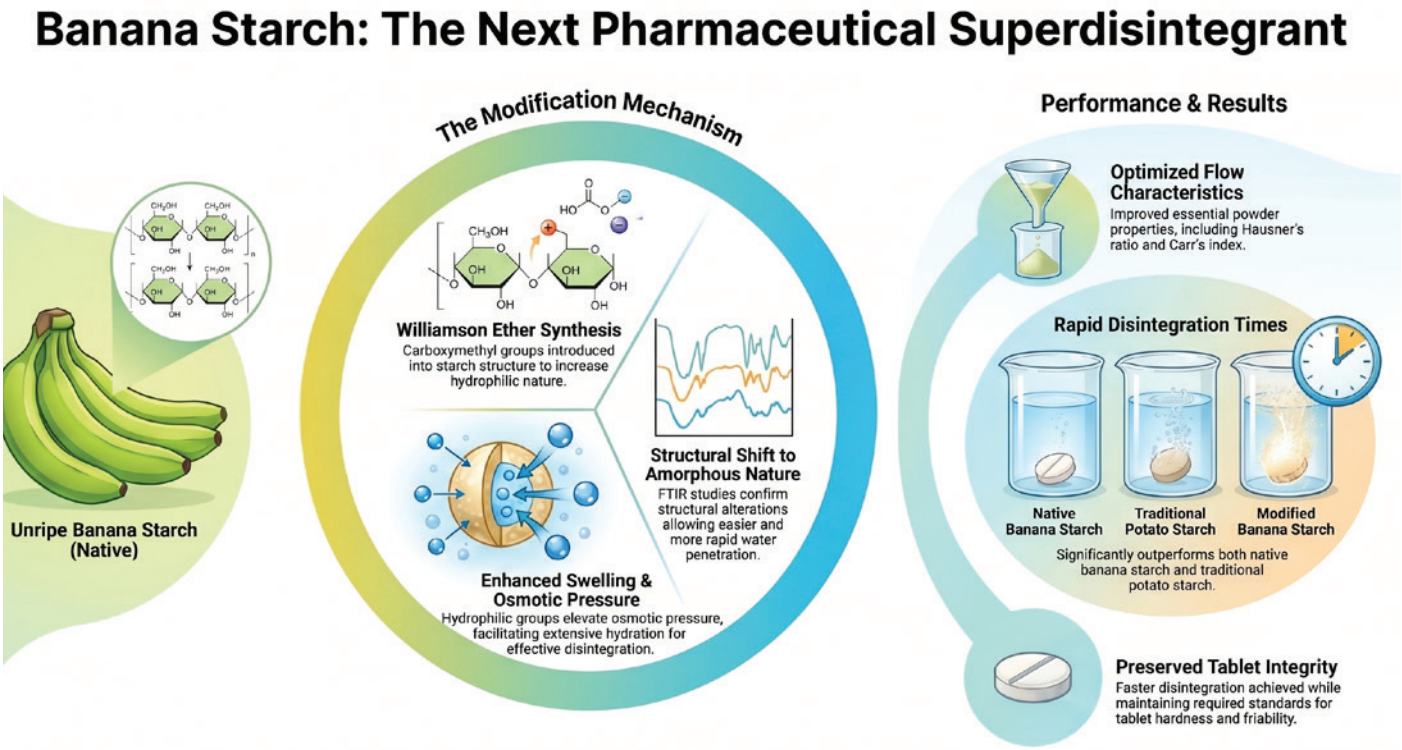
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ABSTRACT

Research Article

Tablets represent the primary choice of oral dosage form for patients. To ensure timely drug release, a tablet must exhibit an appropriate disintegration time, which is typically achieved through the incorporation of various disintegrants. Starch is a widely utilized disintegrant; mostly obtained from natural sources. The unripe fruit of Musa sapientium L. (family Musaceae) (Banana), serves as a rich source of this material for potential pharmaceutical applications. In this study, starch was extracted from bananas, and then chemically modified via Williamson ether synthesis to enhance its disintegration efficacy. The resulting carboxymethylated starch demonstrated superior micromeritic properties, including enhanced flow characteristics, increased solubility and improved swelling capacity compared to native starch. These improvements are attributed to the successful introduction of hydrophilic carboxymethyl groups into the molecular structure of the modified starch. These carboxyl groups induce elevated osmotic pressure and promote extensive hydration, both of which are critical mechanisms for effective disintegration. Differential scanning calorimetry (DSC) and Fourier transform infrared spectroscopy (FTIR) demonstrated that the incorporation of carboxymethyl groups led to structural alterations, increasing the amorphous nature of the modified starch. These changes in material and physical properties of the modified starch facilitated rapid water penetration, accelerate hydration, and enhanced swelling. The functional superiority of this modified excipient was supported by a significant reduction in the disintegration time of diclofenac sodium tablets containing modified starch compared to performance of formulations containing native banana starch or potato starch as the disintegrant. Furthermore, other critical parameters for tablets containing carboxymethylated banana starch, such as hardness, friability, etc., remained within acceptable pharmacopeial limits. These results highlight the potential of carboxymethylated banana starch as a promising disintegrant with possible applications in pharmaceutical formulations.

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KEY WORDS: Banana starch, Williamson ether synthesis, disintegrant, disintegration time, tablet.

INTRODUCTION

Disintegration is one of the key properties influencing the performance of oral tablets. Disintegration is the time taken by the tablet to break apart in fluid into its primary particles which subsequently influences dissolution of the drug. Thus, indirectly the rate of drug release depends on disintegration, and for this to occur, disintegrants are added in the formulation (1). The action of a disintegrant is opposite to that of the tablet binder which functions to hold the powder particles in a compact state. The efficiency of the disintegrant should be balanced with the binding properties of the formulation so that upon disintegration the tablet breaks into granules, followed by formation of finer powder particles. Optimised disintegration performance can be achieved by adding the disintegrant externally and internally during tablet manufacturing (2). When disintegrants come into contact with water they typically undergo processes such as swelling, hydration, change of shape, or even chemical reaction, leading to the breakdown of the tablet. There are many varieties of disintegrants available on the market such as starches, celluloses, gums, clays, ion exchange resins, alginates etc. Starches and celluloses are the most popularly used disintegrants in marketed pharmaceutical formulations.

Starch is the most abundant polysaccharide found naturally in plants. Chemically, it is composed of two glucose polymers-amylose and amylopectin-arranged in semi-crystalline granules whose size, shape, and physicochemical characteristics depend largely on the botanical origin. Because of its biodegradability, biocompatibility, low toxicity, wide availability, and cost-effectiveness, starch has been extensively utilized as a pharmaceutical excipient in the formulation of solid dosage forms. Numerous plant species found abundantly in tropical regions contain starch as a major constituent and have potential applications as pharmaceutical excipients in tablet formulations. One such plant is the unripe fruit of Musa sapientium L. (family Musaceae) commonly known as Banana plant. Banana plant is the largest herbaceous plant in the world, which is widely cultivated in many developing countries (3). Banana fruit is used as a source of starch which is used as a disintegrant in pharmaceutical products. Banana fruits can be consumed all year round and are often used as a nutritional supplement. Considerable physicochemical changes occur in the banana fruit during the ripening process and its post-harvest life span is very short until the point when ethylene is produced. From an industrial point of view, banana starch is associated with some drawbacks, among which are poor thermal stability and retrogradation. Native banana starch has several limitations when exposed to certain processing conditions like heat, change in pH, freezing and thawing, etc. It shows low thermal stability and creates a very sticky paste when heated. This limits its use in food industry. Thus, native banana starch does not possess all required characteristics for specific uses. Converting it into more robust forms requires various different treatments to rearrange starch polymeric chains or insert functional groups thereby modifying the properties of the starch so that it can withstand harsh conditions encountered during food or drug processing. (4) Modification of banana starch can be achieved through physical, chemical, or enzymatic methods. For instance, modifications of starch properties can be carried out by altering environmental conditions, particularly temperature and atmospheric conditions, to improve starch functionality and extend shelf life (5). Chemical modification significantly alters the molecular structure of starch, leading to an increase in its hydrophilic character, which enhances its effectiveness as a disintegrant. Among chemical modification techniques, the Williamson ether synthesis method is a well-established and efficient approach for producing carboxymethylated derivatives. This method enables the introduction of carboxymethyl groups ($–CH_2COO^-$) into the starch molecule, increasing hydrophilicity and promoting rapid water uptake. The modified dry process employed in this technique minimizes the use of organic solvents, making it environment friendly, energy-efficient, and industrially feasible (6).

The present study focuses on the isolation of starch from unripe banana fruits, its chemical modification into carboxymethyl starch using Williamson ether synthesis, and comprehensive physicochemical and material characterization. For evaluating the disintegration potential of the modified starch, formulations were prepared using the model drug diclofenac sodium, a widely prescribed non-steroidal anti- inflammatory drug (NSAID). It is commonly formulated as immediate-release tablets, where rapid disintegration is essential for prompt therapeutic action. The disintegrating efficiency of a diclofenac formulation incorporating modified banana starch was compared with disintegration of formulations based on native banana starch and official potato starch.

One of the main objectives of the present study was to extract starch from the non- conventional source of plantain bananas, Musa paradisiaca Linn, and to explore its possible utilization as a disintegrant. This material was chemically modified to improve its key properties with respect to its sensory, physicochemical, functional and powder properties which were compared with properties of commercially available banana and potato starches and the relative performance of prototype formulations containing diclofenac sodium as the model drug.

MATERIALS

Fresh unripe bananas (Musa paradisiaca) were procured from local markets in Nashik. Sodium monochloroacetate was obtained from the Apex laboratory, Ahmedabad. Ethanol was procured from Modern Science Labs Private limited, Satpur, Nashik. All chemicals were of analytical grade.

METHODOLOGY

Isolation of banana starch

The starch isolation process from banana, as described by Karla Dumancela (7), was adopted with some modifications. Initially, fresh bananas were peeled and chopped into small pieces and soaked in water to prevent any enzymatic browning. The banana pieces were processed using a blender with the addition of distilled water in an appropriate quantity to prepare a slurry. The prepared slurry was filtered through muslin cloth or a fine mesh sieve to isolate the fiber component from the liquid extract. The filtrate was left for a few hours to get a clear supernatant solution with starch sedimentation. The supernatant was discarded, and the sedimented starch was washed repeatedly with distilled water to purify it. The purified starch was dried in an oven to constant weight and powdered for storage purposes.

Modification of starch by using Williamson ether synthesis method

Separated native banana starch was chemically modified using the Williamson ether synthesis method (8, 9). The starch was initially mixed with powdered sodium hydroxide to produce a homogeneous mixture. Ethanol was added in small quantities and the mixture was stirred for 1 hour to ensure adequate activation. Sodium monochloroacetate (SMCA) was then added into the mixture at room temperature with constant stirring. After the reaction was complete, the mixture was filtered through Whatman filter paper and washed with aqueous ethanol (50 %v/v) to remove any unreacted material. The purified carboxymethylated starch was subsequently dried in a hot air oven at 45-50 °C until a constant weight was obtained. (See equation above)

$$\text{Starch–OH} + \text{ClCH}_2\text{COONa} \xrightarrow[\text{Ethanol}]{\text{NaOH}} \text{Starch–O–CH}_2\text{COONa} + \text{NaCl}$$

Determination of degree of substitution

The degree of substitution (DS) represents the average number of carboxymethyl groups replaced per anhydroglucose unit (AGU) of starch. Theoretically, the DS ranges from 0 to 3, as there are three hydroxyl groups per AGU available for substitution. It was assessed by the back-titration technique, as follows:

Modified banana starch (1.0 g) was accurately weighed and transferred to a conical flask containing 50 mL of 75% (v/v) ethanol. The flask was secured with a stopper and the suspension was agitated in a water bath at 50 ± 2 °C for 30 min and then was allowed to cool at room temperature. Then, 40 mL of 0.5 N potassium hydroxide (KOH) solution was added in the flask and it was again sealed with a stopper and then agitated continuously (at room temperature) for 48 h on a rotary flask shaker to complete the saponification of the carboxymethyl groups. Excess alkali was determined by titration against 0.5 N hydrochloric acid (HCl) using phenolphthalein as the indicator. A blank determination was performed in absence of the modified banana starch under the same experimental conditions.

The percent carboxymethyl group added to the starch was calculated using Equation (1):

$$\%\ carboxmethyl = \frac{(B - S) \times N \times 5.8}{weight\ of\ sample} \qquad (1)$$

Where, B = blank titration volume (mL) of HCl, S = sample titration volume (mL) of HCl, N = Normality of HCl, 5.8 = conversion factor for carboxy methyl group

The degree of substitution was determined using Equation (2):

$$DS = \frac{162 \times \%\ carboxmethyl}{7400 - (74 \times \%\ carboxymethyl)} \qquad (2)$$

Here, 162 = molecular weight of AGU, 7400= 100 times the molecular weight of carboxymethyl group, 74= molecular weight of carboxymethyl group

Characterization of native banana starch and modified starch

Organoleptic Properties (10)
Modified banana starch and native banana starch were evaluated for organoleptic characteristics such as colour, odour, and appearance.

Identification Test (11)
Iodine test
Preparation of Iodine solution
Iodine solution was prepared by first dissolving 50 mg of potassium iodide in distilled water. Iodine crystals (25g) were added and stirred until all the iodine was dissolved and the volume of the solution was made up to 1000 ml using distilled water.

Preparation of and testing of Starch Mucilage
A starch mucilage was prepared by triturating 1 g of soluble starch with 5 mL of water. This was gradually added, with continuous stirring, to sufficient water to produce 100 mL of dispersion. The mixture was boiled for a few minutes, cooled, and then was filtered.

'To test the mucilage, a few drops of iodine solution were added to the filtrate. A characteristic colour change to blue confirmed that starch was present in the mucilage.

Solubility (11, 12)
Three samples were prepared by suspending 0.6 g of starch in 40 mL of distilled water in separate beakers and was then covered with aluminium foil to avoid water loss. These suspensions were then heated at 60°C, 70°C and 80°C respectively for 30 minutes. They were continuously stirred to avoid formation of lumps. The resulting suspensions were then centrifuged at 3000 rpm for 15 minutes. The supernatant was separated from the sediment of each sample, and the weight of the sediment was recorded. The supernatant was transferred to a dry petri dish of known weight and was dried at 130 °C for two hours. The dried residue was determined as the amount of soluble starch (Wss), and the solubility of starch in percent was determined using Equation (3):

$$Solubility = \frac{Wss}{Ws} \times 100 \quad (3)$$

Where, Wss = wt of soluble starch (gm) and Ws = wt of starch sample (gm)

Ash Value (13)
A clean and dry crucible was weighed, and its weight was noted as X g. Approximately 2 g of the sample was weighed into the crucible and the total weight was noted as Y g. The sample was first heated gently until no smoke was produced, and then it was burned in a muffle furnace at 600 °C for 2 hours. After burning completely, the crucible was allowed to cool in a desiccator and then weighed again, and its weight was noted as Z g. Ash value was calculated using Equation (4):

$$Ash\ value\ content = \frac{(Z - X)}{(Y - X)} 100 \quad (4)$$

Water Binding Capacity (WBC) (14)
About 1.0 g of starch sample was added to 5 ml of water in a centrifuge tube. This was shaken for 1 min and then was centrifuged at 3000 rpm for 10 min at room temperature. The supernatant was decanted and the weight of the residue obtained was measured. Water binding capacity of the starch sample was calculated using Equation (5):

$$WBC = \frac{Weight\ of\ residual\ starch}{Initial\ weight\ of\ starch\ sample} 100 \quad (5)$$

Swelling Index (15)
0.5 g sample of starch was mixed separately with 25mL of different media such as 1N NaOH, 1N KOH, 1N HCL, distilled water, and 1N NaCl in separate beakers. The mixtures were heated in a water bath at 60 °C for 30 min and were allowed to cool. During the cooling period, the starch settled at the bottom of the cylinders. The supernatant was decanted and the weight of the swollen residue was determined. Swelling index of starch in each medium was determined using Equation (6):

$$Swelling\ index = \frac{Weight\ of\ residue}{Weight\ of\ dry\ sample} \quad (6)$$

Moisture Content
5.0 g of sample was accurately weighed in a pre-dried and tared petri dish. The sample was dried in a vacuum oven at 120 °C for 4 hours under reduced pressure (<100 mmHg). The dish was cooled in a desiccator for 30 min and was then re-weighed. The amount of moisture present was calculated using Equation (7):

$$\%\ moisture\ content = \frac{Sample\ weight\ (g) - dry\ sample\ (g)}{Sample\ wt.\ (g)} \times 100 \quad (7)$$

pH Measurement (8)
About 5.0 grams of starch was dispersed in 25 mL of carbon dioxide (CO_2)-free distilled water and stirred for 1 minute. The suspension was allowed to stand for 15 minutes after which the pH was measured using a calibrated pH meter (Toshniwal instrument mfg. pvt.ltd. Ajmer, CL54+).

Fourier Transform Infrared Spectroscopy (FTIR)
FTIR analysis of the starch samples was carried out using a FTIR spectrometer (Cary 630, Agilent Technologies). Powdered samples were prepared using potassium bromide (KBr) pellets by dispersing the samples in dry potassium bromide. Spectra were obtained in the region of 400 cm^{-1} to 4000 cm^{-1}.

Scanning Electron Microscopy (SEM)
Morphology of native and carboxymethyl starch was studied using a scanning electron microscope (JSM-200, JEOL, Tokyo, Japan). Samples were placed on metal stubs and coated with gold for better conductivity. The images were taken under an accelerating voltage of 10 kV and magnification of 500X.

Differential Scanning Calorimetry (DSC)
Thermogravimetric properties such as gelatinization temperature and melting point were determined using a DSC apparatus (Mettler Toledo, Mumbai, India). About 5.0 mg of each sample was placed in aluminium pans and heated from 30 °C to 300 °C. Endothermic and exothermic peaks were observed.

Powder X-ray Diffraction (PXRD)
PXRD patterns of samples were recorded using an X-ray diffractometer (MAXima X XRD-7000, Shimadzu, Japan) operated at 40 kV and 30 mA with Cu Kα radiation (λ = 1.54 Å). The data were acquired in the 2θ range of 10°–40° at a scanning speed of 4°/min and step size of 0.02°.

Formulation and manufacture of diclofenac tablets
Tablets of diclofenac sodium were prepared containing various starches as disintegrants in formulations outlined in Table 1. Tablets were prepared using direct compression on a six station rotary tablet machine (Rimek mini press) with 9 mm flat-faced punches.

Characterization of tablet blends
The powder blends of all formulations were evaluated for flow properties, including bulk density, tapped density, angle of repose, compressibility index, and Hausner's ratio (16).

Bulk density: The bulk density of the powder was determined by accurately weighing a known quantity of powder (30gm) and transferring it into a clean, dry 100 ml graduated measuring cylinder without compacting. The initial volume occupied by the powder (bulk volume) was recorded, and bulk density was calculated as the ratio of the mass of the powder to its bulk volume (Equation 8).

$$\text{Bulk density } (\rho b) = \frac{Mass}{bulk\ volume} \quad (8)$$

Tapped Density
The tapped density of the sample was determined using the same sample in the graduated cylinder as used for bulk density testing. The cylinder was tapped for 100, 150 and 200 taps at a constant rate until there was no further decrease in volume. This volume was recorded as the tapped volume, and the tapped density was calculated as the ratio of the weight of the powder to the tapped volume (Equation 9).

Table 1. Formula for formulations of diclofenac tablets using various disintegrants

Ingredient	F1 (mg)	F2 (mg)	F3 (mg)	F4 (mg)	F5 (mg)	F6 (mg)
Diclofenac sodium (API)	50	50	50	50	50	50
Native banana starch (Disintegrant)	20	25	-	-	-	-
Modified banana starch (Test Disintegrant)	-	-	20	25	-	-
Potato starch (Disintegrant)	-	-	-	-	20	25
Talc (Glidant)	3	3	3	3	3	3
Magnesium stearate (Lubricant)	2	2	2	2	2	2
Lactose monohydrate (Diluent)	150	145	150	145	150	145
Total	225	225	225	225	225	225

$$\text{Tapped bulk density } (\rho t) = \frac{Mass}{\text{tapped volume}} \quad (9)$$

Angle of repose

Powder flow was determined by measuring the angle of repose. Powder was made to flow through a funnel set at a standard height of 10 cm above a levelled surface, to form a cone of material. The height (h) and the radius (r) of the cone were measured, and the angle of repose was calculated (Equation 10)

$$\text{Angle of repose } \theta = \tan^{1}(h/r). \quad (10)$$

Carr's index

The Carr's index, which is also a measure of the flowability of a powder sample, was calculated using the bulk density (ρb) and tapped density (ρt) (Equation 11)

$$\text{Carr's index} = (\rho t - \rho b)/\rho t \times 100 \quad (11)$$

Hausner Ratio (HR)

The Hausner ratio (HR) was determined using the ratio of tapped density and bulk density (Equation 12)

$$HR = \rho t / \rho t \quad (12)$$

Characterization of tablets

Tablet dimensions (17)

Tablet dimensions were measured using a Vernier calliper (For-Bro Engineers, India) with a sample of ten randomly selected tablets.

Weight variation (18)

Twenty tablets were randomly selected and weighed individually. The individual weights were compared with the calculated mean weight. According to pharmacopoeial limits, not more than two tablets should deviate by more than ± 5 % of the mean weight.

Tablet hardness

Tablet hardness was measured using a Pfizer hardness tester. Tablets of each formulation were individually tested to determine their resistance to mechanical stress. The hardness was expressed in terms of kg/cm².

Friability testing

Friability testing of tablets was performed using a friabilator (Erweka, TAP, Germany). A pre-weighed batch of the tablets (6 tablets) (Iw) was placed inside the plastic chamber of the friabilator which was operated at 25 rpm for 4 minutes. During the test, the tablets were subjected to abrasion as well as shock as each tablet fell from a height of 6 inches during each revolution of the chamber. After the test was completed, tablets were removed, dusted off using a soft muslin cloth, and weighed again (Fw). Friability of tablets were calculated by using equation 13. Results were compared to the pharmacopoeial limits. Based on the requirement in the Indian Pharmacopoeia, a loss in weight of less than 1% is acceptable.

$$F = (Iw - Fw)/Iw * 100 \quad (13)$$

where, Iw is initial weight of tablets, Fw is final weight of tablets.

Disintegration time

The disintegration time of the tablets was tested using a standard disintegration test apparatus maintained at 37 ± 0.5 °C (Tablet disintegration test apparatus (double unit), Labline.). The time for complete disintegration of tablets was recorded. Complete disintegration of the tablets occurred when no tablet core could be palpated in the basket.

RESULTS AND DISCUSSION

Physicochemical characterization of banana starch and modified banana starch

Organoleptic characterization

The organoleptic and physicochemical properties of native and chemically modified banana starch are tabulated in Table 2. The two starches were both acquired in powder form and had no odour and no distinct taste. Native starch was observed to be white while chemically modified starch was found to be brownish white.

When tested using iodine solution, both native and chemically modified banana starches caused the solution to turn blue, indicative of starch-containing material.

Ash value

Ash value reflects the amount of inorganic, non-combustible residue (minerals) present in starch and is an indicator of its purity. According to the literature, the ash value of pure starch is typically less than 1 % (13, 19, 20). In this study, the ash

Table 2. Characterization results for banana starch and modified banana starch

Test	Results for Native banana starch	Results for Modified banana starch	Results for Standard Specifications
	Organoleptic properties		
Form	Powder	Powder	Powder
Colour	White	Brownish	Brownish white
Odor	Odourless	Odourless	Odourless
Taste	Tasteless	Tasteless	Tasteless
	Identification tests		
Sensitivity to iodine	Blue Colour	Blue Colour	Blue Colour
Starch mucilage	Blue colour	Blue colour	Blue colour
Ash value (%)	1.12 ± 0.05*	0.89 ± 0.02*	< 1 %
Moisture content (%)	11.72 ± 0.11*	7.25 ± 0.16*	8-14 %
pH value	5.31 ± 0.04*	6.21 ± 0.02*	4-7
Water binding capacity (%)	130.65 ± 4.92*	437.66 ± 9.80*	

*Mean ± S.D. (n=3)

value of banana starch was found to be higher (1.12 %), suggesting the presence of residual, non-burnable materials, likely due to incomplete removal of banana fruit components during isolation. However, after chemical modification, the ash value decreased to 0.89 ± 0.02 %, indicating improved purity and a reduction in mineral content.

Moisture content and pH

The moisture content of banana starch is typically reported in the range of 8–14 % (12). Presence of moisture influences the physicochemical properties of starch. Higher moisture levels enhance interparticle binding between starch granules, thereby reducing flowability. In the present study, the moisture content of both native and modified banana starch samples was found to be less than 12 % (Table 2), with modified banana starch exhibiting significantly lower moisture content than the native starch. Such reduction in moisture content is expected to enhance the flow properties and water-binding capacity of the carboxymethylated starch.

The pH of native banana starch was found to be 5.31, indicating a slightly acidic nature, whereas the modified starch exhibited a higher pH of 6.21. This increase in pH following modification suggests the successful incorporation of carboxymethyl groups into the starch structure which was confirmed by FTIR. The observed shift towards near-neutral pH can be attributed to the presence of carboxymethyl substituents predominantly in their sodium salt form ($–COO^-Na^+$). This modification alters the acid-base characteristics of the starch, indicating effective chemical modification and contributing to changes in its physicochemical properties.

Solubility and swelling capacity

The solubility and degree of swelling of modified starch were found to be higher than those of native banana starch as represented in Table 3 and Figure 1. These observations are consistent with earlier reports indicating that carboxymethyl starch exhibits enhanced solubility and swelling capacity due to the presence of hydrophilic carboxymethyl groups (21 – 23). These groups enhance water uptake by promoting the

Figure 1. Degree of swelling of Native banana starch and Modified banana starch

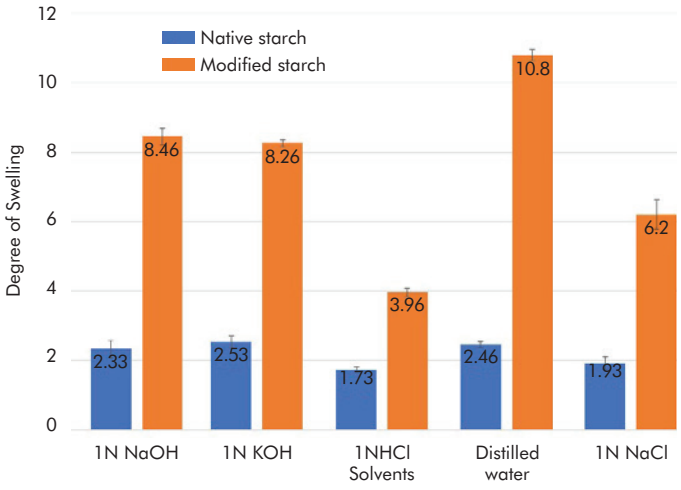


Table 3. Solubility of native banana starch and modified banana starch at different temperatures

Temperature (°C)	Solubility (g/100ml)	
	Native Starch	Modified Starch
60	1.32 ± 0.02	16.09 ± 0.47
70	5.32 ± 0.06	22.08 ± 0.51
80	8.20 ± 0.07	33.02 ± 0.19

formation of hydrogen bonds with water molecules, thereby facilitating greater water retention and swelling capacity.

Solubility of both native and modified starch increased with increased temperature and at any particular temperature, the solubility of modified starch was found to be higher than native starch.

Water binding capacity

Water binding capacity (WBC) is a critical parameter influencing swelling behaviour and consequently, disintegration efficiency. An increase in WBC promotes greater hydration, leading to improved swelling and faster tablet disintegration (24). In the present study, modified banana starch exhibited a significantly higher WBC (437.66 ± 9.80 %) as compared to native starch (130.65 ± 4.92 %). This marked enhancement in water uptake can be attributed to the presence and ionization of hydrophilic carboxyl groups in the modified starch, which elevate osmotic pressure and facilitate extensive hydration. The substantially higher WBC of the modified starch indicates its superior swelling ability and supports its potential as an effective pharmaceutical disintegrant.

Flow properties of starch

Flow properties of powders are influenced primarily by particle size, shape, and surface characteristics (25, 26). In the present study, the flow behaviour of modified banana starch was evaluated using angle of repose, Carr's index, and Hausner ratio. The modified starch exhibited improved flow properties compared to native starch (Table 4). A Carr's Index value of <10 % is an indication of excellent flow, 11–15% indicates good flow and >26% indicates poor flow. An angle of repose of < 25° indicates good to excellent flow, while >40 ° indicates poor flow. A Hausner Ratio of <1.12 indicates excellent flow, 1.12–1.18 indicates good flow, and >1.35 indicates poor flow properties (18).

The observed improvement in flow properties of the modified starch suggests alterations in particle size distribution and morphology following chemical modification. Such modification is likely to improve particle surface characteristics, reducing interparticle friction and cohesion. Consequently, the enhanced flow behaviour highlights its improved handling properties and suitability of the modified starch for direct compression applications in pharmaceutical formulations.

Structural characterization results

Degree of Substitution

The degree of substitution (DS) is described as the average number of substituent groups on each anhydro-glucose unit (AGU), which is the fundamental unit of the starch molecule. Since there are three hydroxyl groups in each AGU, the theoretical range for the DS is between 0 and 3. The level of DS profoundly affects the physical properties of starch, including its water solubility, which is positively correlated with the DS value (22, 23).

In this research, the DS of the substituted banana starch was estimated to be 1.71 ± 0.02 indicating high substitution. It means that the substituent groups were efficiently incorporated in the starch structure, thereby improving its interaction with water molecules. In addition, the modified starch shows better solubility and swelling ability due to the increased interaction with water, facilitating its application as a potential pharmaceutical disintegrant.

Fourier Transform Infrared (FTIR) spectroscopy

FTIR spectra of the native banana starch as shown in Figure 2 and carboxymethylated banana starch as shown in Figure 3

Table 4. Flow properties exhibited by native banana starch and modified banana starch

Sample	Angle of repose	Carr's index	Hausner ratio
Modified starch	24.04 ± 0.33°*	8.28 ± 1.37 %*	1.09 ± 0.01*
Inference	Excellent flow	Excellent flow	Excellent flow
Native starch	29.04 ± 0.31°*	12.93 ± 2.27 %*	1.14±0.03*
Inference	Good flow	Good flow	Good flow

*Mean ± S.D. (n=3)

Figure 2. FTIR spectrum of native banana starch

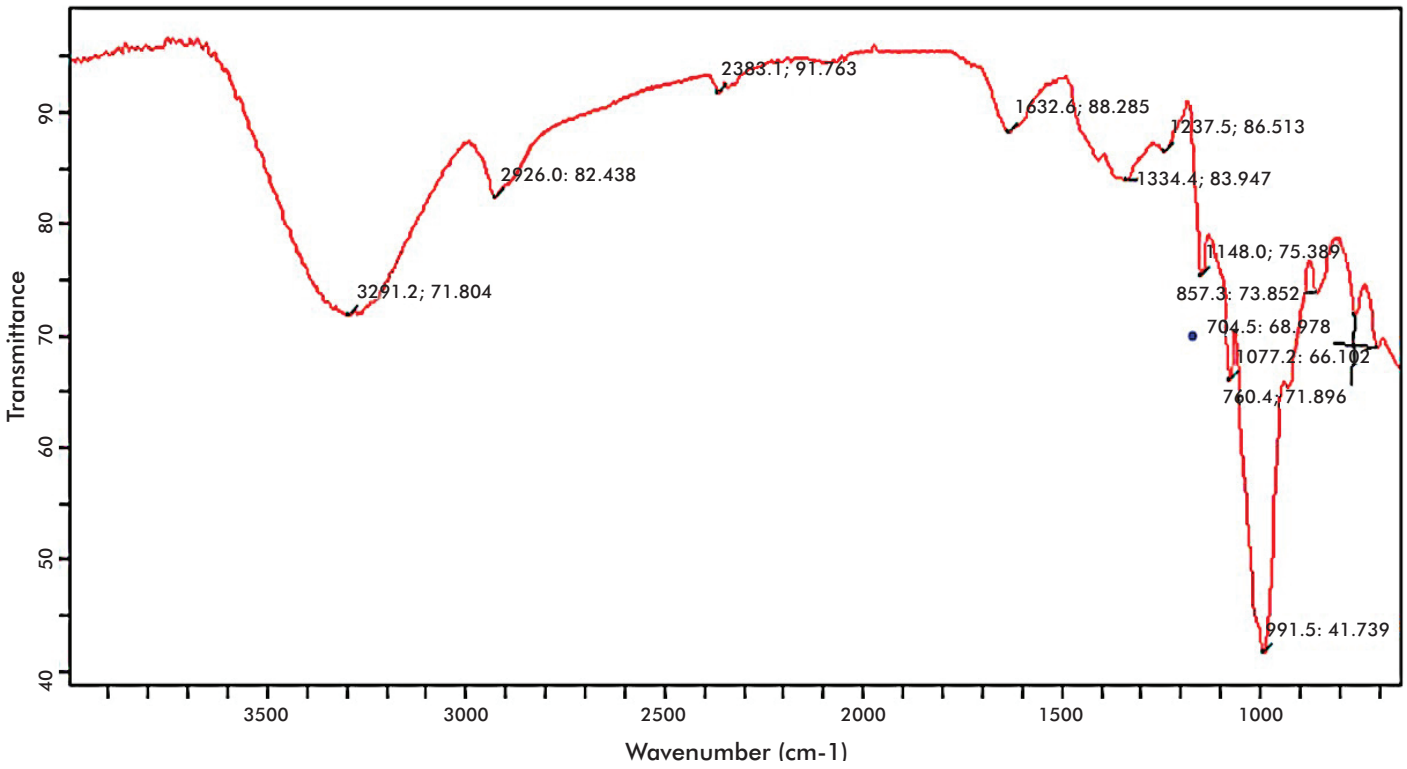
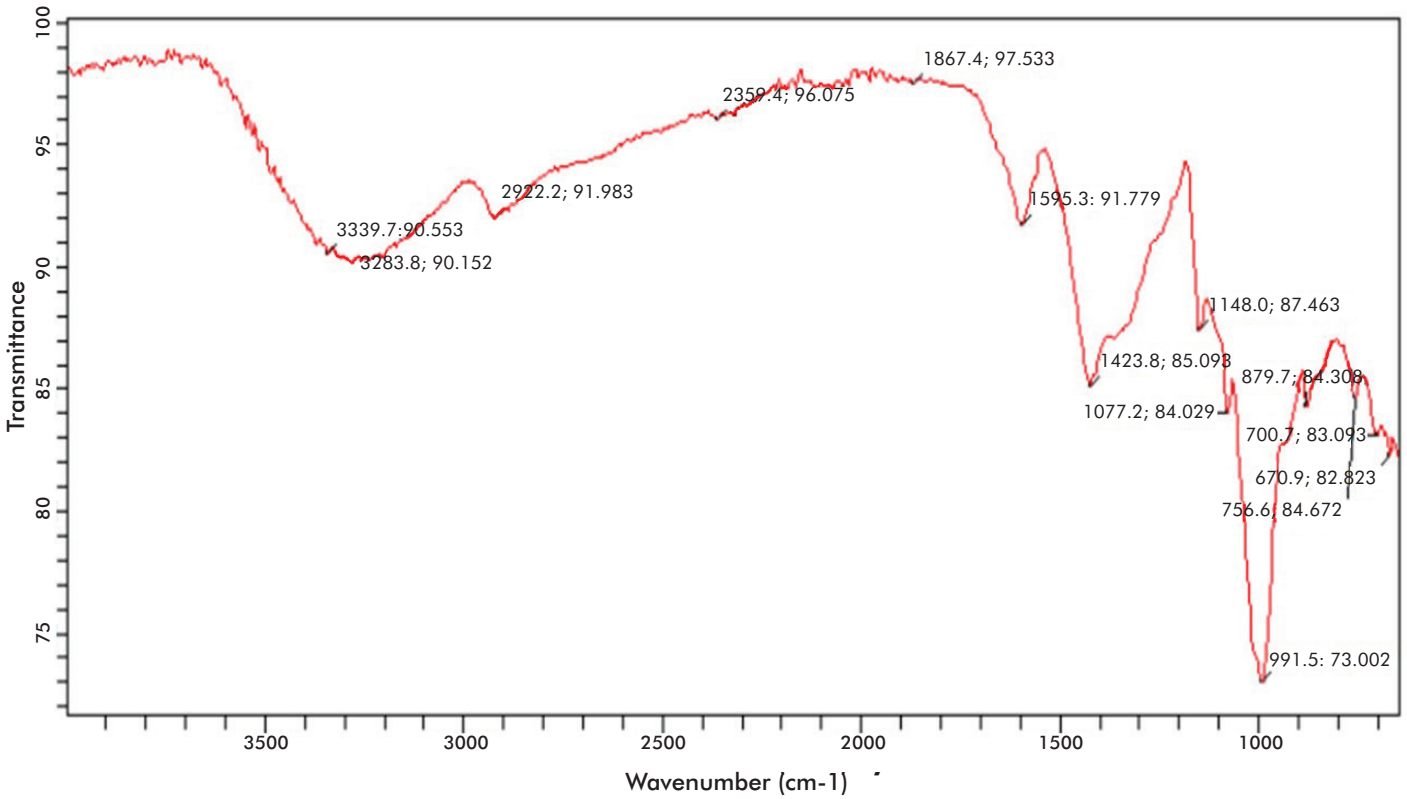
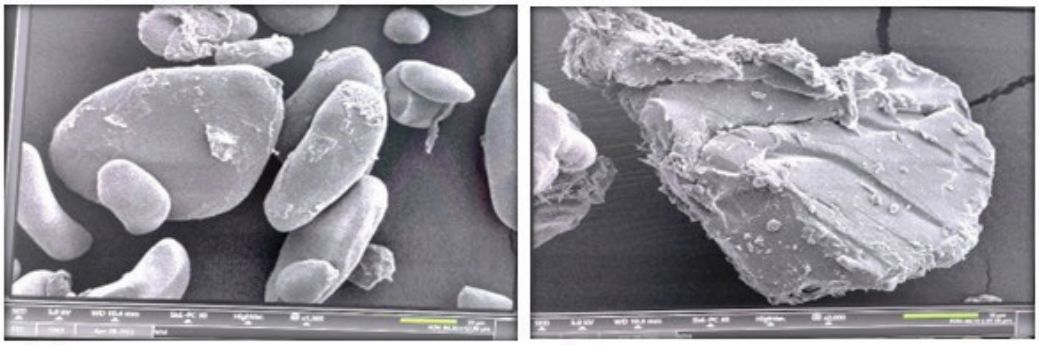


Figure 3. FTIR spectrum of modified banana starch



(with DS = 1.71-1.73) highlight the structural changes in the modified starch and corroborated the results on the degree of substitution. There were absorption bands in the range of 3600-3000 cm^{-1} (native: 3251 cm^{-1}; modified: 3339-3283 cm^{-1}), indicating O-H stretching vibrations related to hydrogen bonding. The slight change in position and intensity of the O-H absorption band of the modified starch suggested that the hydrogen bonding was influenced by the introduction of the substituent groups.

Figure 4. SEM images: Native banana starch vs Modified banana starch



A Native banana starch **B** Modified banana starch

The absorption bands at ~2920 cm^{-1} in both samples relate to C-H stretching vibrations of the glucose units. An additional band at ~2359 cm^{-1} in the spectrum of the modified starch can be related to C-H stretching vibrations and suggests the presence of the introduced carboxymethyl group.

One of the most distinctive differences in the spectra is the presence of strong absorption bands in the region of ~1595-1423 cm^{-1} in the spectrum of the modified starch. It is related to the asymmetrical and symmetrical COO^- stretching vibrations of the carboxymethyl group. This proves that the carboxymethyl group was successfully introduced into starch structure

Scanning Electron Microscopy (SEM)

The native banana starch had granules that were ellipsoidal in shape with a smooth surface morphology. In comparison, the modified starch had significant morphological changes, which included cracks on the surface of the granules, roughness and irregularity of the surface structure.

These morphological differences signify the alteration of the granule structure after modification of the starch. The increase in the roughness of the surface and creation of fissures provides increased effective surface area and consequently higher water penetration (Figure 4). This aligns with results of the swelling index and water binding capacity (11), (15) that are related to the higher water penetration that leads to faster swelling and improved disintegration.

Differential Scanning Calorimetry

The results of the DSC thermograms of native starch and modified banana starch are shown in Figures 5 and 6, respectively. The endothermic peak in the temperature range of 50 to 126 °C is due to moisture loss and starch gelatinization (peak at 82.79 °C). The thermal event at ~206 °C is associated with the initiation of normal degradation of starch polymer chains. Modified banana starch with lower peak gelatinization temperature of 78.25 °C and end set temperature at 116.14 °C indicates compromised crystallinity of the starch structure due to addition of carboxymethyl groups that reduce the hydrogen bonding capacity. As a result, the modified starch was more amorphous in nature. In other words, the ordered crystalline structure of native starch was destroyed by the substitution of hydroxy groups with hydrophilic carboxymethyl groups, which reduced the intermolecular hydrogen bonding and increased the accessibility of water to the granules. This results in the substituted starch gelatinizing more easily at lower temperatures (27).

This physical change in modified banana starch made it easier for water to penetrate the starch particles and to hydrate them, and swell facilitating its properties as a potential disintegrant. The degradation peak shifted to a higher temperature of 225.82 °C as compared with the native starch, indicating that the ether linkages formed in the Williamson ether synthesis improve the thermal stability of the modified polymer. The higher enthalpy of degradation (–147.78 J/g) is additional evidence of the modified molecular interactions due to chemical substitution.

Powder properties of diclofenac tablet formulations

Powder blends containing modified banana starch exhibited better flow properties than those containing native banana starch and potato starch. Formulations containing the modified banana starch showed lower angle of repose, Carr's index, and Hausner's ratio which indicate excellent compressibility and flowability, essential for ensuring uniform

Figure 5. DSC of native banana starch

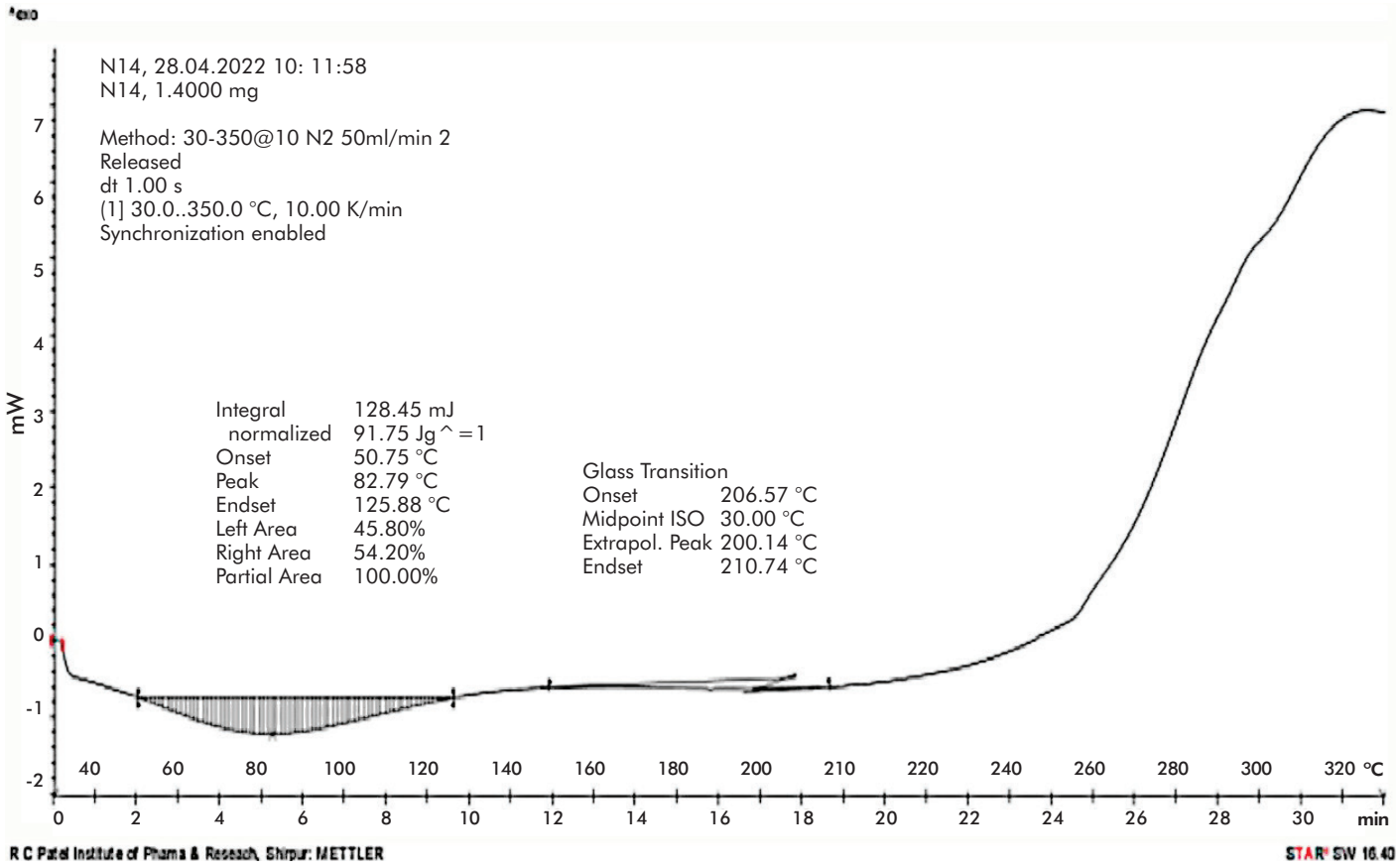


Figure 6. DSC of modified banana starch

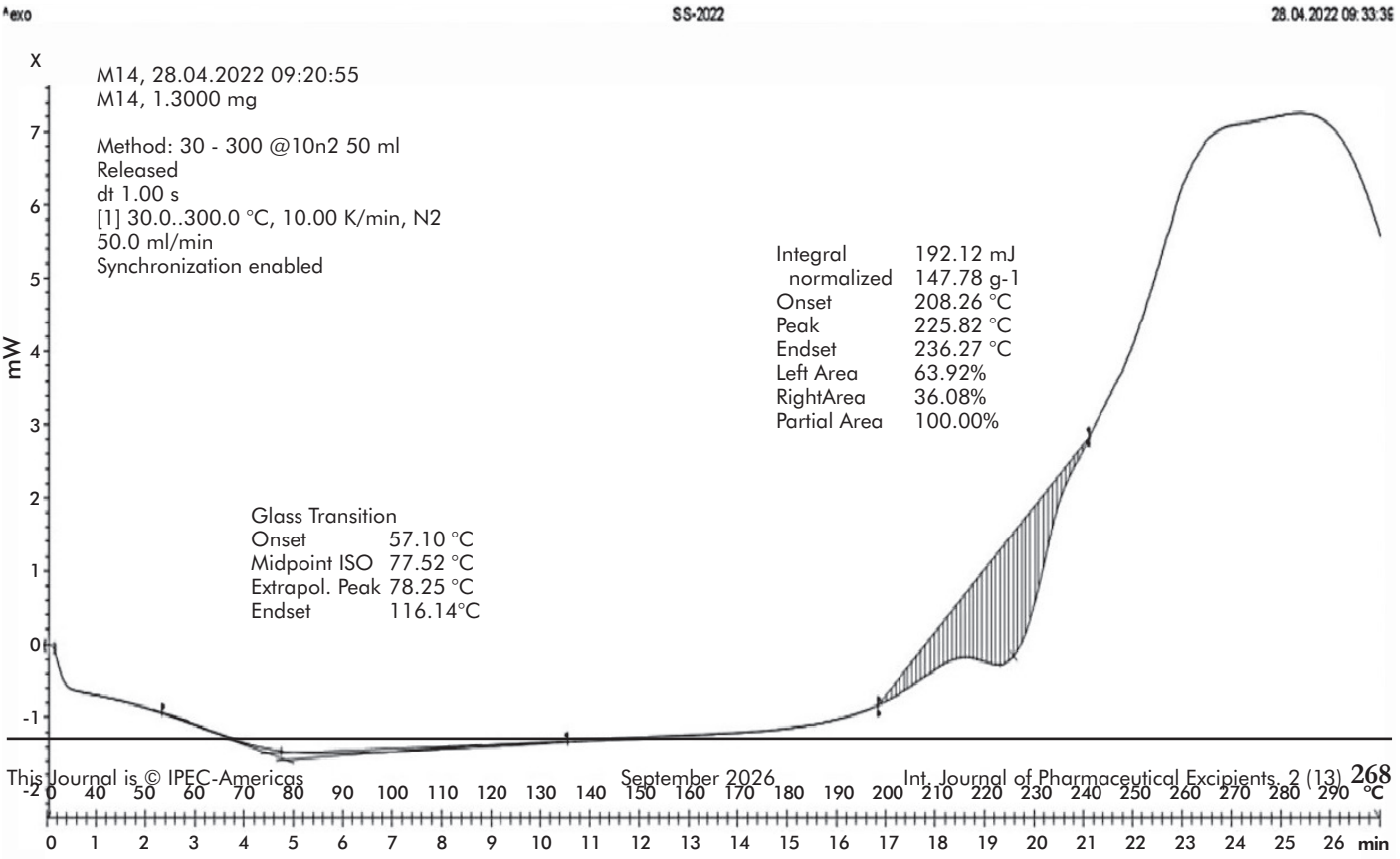


Table 5. Powder properties of tablet formulations

Formulation code	Angle of repose (°)*	Bulk density (g/cm³) *	Tapped density* (g/cm³)	Carr's Index (%)*	Hausner ratio*	Inference
F1	29.22 ± 0.95	0.40 ± 0.014	0.47 ± 0.016	8.69 ± 4.11	1.15 ± 0.05	Good flow
F2	29.25 ± 1.57	0.40 ± 0.012	0.48 ± 0.020	13.33 ± 3.35	1.18 ± 0.04	Good flow
F3	24.91 ± 0.52	0.52 ± 0.016	0.58 ± 0.021	3.57 ± 4.31	1.10 ± 0.05	Excellent flow
F4	24.95 ± 0.90	0.46 ± 0.012	0.52 ± 0.009	6.00 ± 4.67	1.11 ± 0.06	Excellent flow
F5	28.13 ± 0.75	0.42 ± 0.028	0.54 ± 0.012	21.42 ± 5.81	1.30 ± 0.10	Passable flow
F6	28.06 ± 0.47	0.38 ± 0.009	0.48 ± 0.016	13.04 ± 6.17	1.27 ± 0.09	Good flow

*Mean ± S.D. (n=3)

Table 6. Performance test results for formulations F1 to F6

Formulation code	Average weight (mg)*	Thickness (mm)*	Hardness (kg/cm2)*	Friability (%)*	Disintegration time (min)#
F1	225.5 ± 0.67	3.33 ± 0.06	3.1 ± 0.16	0.71 ± 0.01	4.00 ± 0.16
F2	225.54 ± 0.94	3.34 ± 0.09	3.3 ± 0.21	0.79 ± 0.02	4.28 ± 0.08
F3	224.31 ± 1.22	3.00 ± 0.37	3.06 ± 0.08	0.45 ± 0.02	3.23 ± 0.08
F4	225.2 ± 1.03	3.14 ± 0.31	2.51 ± 0.12	0.41 ± 0.01	3.40 ± 0.04
F5	226.2 ± 0.65	2.83 ± 0.44	2.80 ± 0.08	0.84 ± 0.01	4.15 ± 0.25
F6	224.5 ± 0.42	3.33 ± 0.13	2.60 ± 0.16	0.98 ± 0.02	4.11 ± 0.06

*Mean ± S.D. (n=3) #Mean ± S.D.(n=6)

tablet compression with uniform weight. Formulations containing native and potato starch showed comparatively inferior flow, which may lead to weight variation and poor die filling during compression (Table 5).

Testing of manufactured tablets:

Diclofenac tablet formulations (F1–F6) were compressed and subsequently evaluated for thickness, hardness, friability, and disintegration time to facilitate a comparative assessment among the formulations (Table 6).

All the tablet formulations complied with pharmacopoeial specifications for weight variation, thickness, hardness, and friability (28). Tablets formulated with modified banana starch exhibited a shorter disintegration time as compared to those containing other starches. This observation is consistent with its improved physicochemical properties, particularly the swelling index and water binding capacity, thereby supporting the effectiveness of the modified banana starch as a possible pharmaceutical disintegrant.

CONCLUSION

Carboxymethyl banana starch was successfully prepared through Williamson etherification of native banana starch. The modified starch had improved properties, such as lower ash content (purity), lower moisture content, as well as higher pH due to the presence of carboxymethyl groups in the sodium form. More specifically, it showed, improved solubility, swelling power, and water retention ability, coupled with a high degree of substitution (DS = 1.71) and improved disintegrant properties. Changes in its molecular structure and morphology were confirmed through FTIR, SEM, and DSC techniques. The observed increase in roughness and formation of fissures likely increased the surface area of the material, facilitating easy access of water. The lowered gelatinization temperature indicates reduced crystallinity and hydrogen bonding which align with its improved solubility, water binding and disintegration.

Formulation results showed that diclofenac tablets containing the modified banana starch exhibited much shorter disintegration time than those prepared using conventional starches. The modified banana starch also demonstrated acceptable flow properties that are critical for direct compression and efficient manufacturing of tablet dosage forms. Overall, the current study demonstrates that carboxymethylated banana starch represents a promising excipient material with potential as an efficient disintegrant for pharmaceutical formulations.

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