

Studies on Synergistic Effects of Thermal Treatment on Physicochemical Properties of Starch Blends

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Abstract

Aim: The aim of the study was to evaluate the potential synergistic effect of natural and thermally modified starch blends from maize and potato. **Materials and Methods:** The maize and potato starches were combined in proportions of 1:1 (NSB1) and 1:2 (NSB2) and thermally treated (pregelatinization and retrogradation). For their application as excipients, these thermally treated starch blends were compared to natural starch blends for physicochemical parameters such as moisture content, water holding capacity, swelling and solubility, and amylose concentration. **Results and Discussion:** The amylose content of the heat-treated gums increased, indicating that it could be used in colon medication administration. The increase in water holding capacity from $218.13 \pm 0.13\%$ to $732.27 \pm 0.34\%$ demonstrates its promise in hydrogel creation. The moisture percentage of all the blends was in the range of $10.10 \pm 0.03\%$ – $15.42 \pm 0.03\%$, which were well within the range specified in Indian Pharmacopoeia. All of the samples' pH levels were determined to be mildly basic (7.15–7.46). **Conclusion:** The potato and maize modified starch blends demonstrated a promising synergistic impact compared to native blends as an adjuvant in the formulation of various drug delivery systems.

Key words: Physicochemical properties, pregelatinization, retrogradation, starch blends

INTRODUCTION

Starch is a naturally occurring polysaccharide available abundantly in plants where it is stored in the form of energy in the parts such as root tubers, corms, and seeds. They are known as the major source of carbohydrates in the diet of human.^[1] Amylose as well as amylopectin are the chief chemical constituent of the starch. Both amylose and amylopectins consists of α -D-glucose units connected through α -1,4 and α -1,6 linkages. Amylose is structurally a linear polymer, while amylopectin is predominantly branched through α -1,6 linkages and larger.^[2] Starch finds its place in various foods as well as in pharmaceutical industries due to its useful functional properties. However, the native starches are difficult to use in pharmaceutical industries due to their suboptimal gelling properties, low sheer resistance, and higher freeze thawing capability. Hence, preferably chemical modifications like carboxymethylation, esterification can provide solution to these problems. However, the uses of chemical modifications were diminished due to problems of toxicity. As per the general trends on research leaned on more natural methods for

alternatives to chemical modifications, one possibility found to improve the functional properties of native starch is the use of blends of different native and/or physically modified starches. The blended starches were shown to improve gelation and resist syneresis. Hence, the present work explores the impact on physicochemical properties due to thermal treatment of maize and potato starch blends.^[3]

MATERIALS AND METHODS

Materials

The procurement of both maize as well as potato starches was done from Central Drug House, India. Throughout the

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whole study, the analytical grade materials were used which were procured from Spectrochem and Rankem. The various solutions were prepared using Millipore water.

Blending of starch

Starch blends were prepared by weighing maize and potato starches in two proportions and mixed in ratios of 1:1 (NSB 1) and 1:2 (NSB2). The blended starches were sieved using 0.841 mm sieve and further mixed using a mechanical blender (IKA A11, Germany).^[4,5]

Preparation of pregelatinized starch

The pregelatinization of blended was carried out as described by Deepika *et al.*, with slight variation. A quantity of 10 g NSB1 and NSB2 samples were suspended in Millipore water (1:10 w/v) and heated in water bath at 90°C for different time intervals of 15, 20, and 25 min (PSBA1, PSBA2, PSBA3 for NSB1 PSBB1, PSBB2, and PSBB3 for NSB2) continued with constant agitation (RemiMLH1, India) at 700 rpm. The resulting paste was dried at 50°C for 24 h in a hot air oven (Scientific Corporation, India) and pulverized in an analytical mill (Ika A-11, Germany) and passed through a sieve with the mesh size of 125 µm and stored in air tight container till further use.^[6]

Retrogradation of starch

Starch suspensions (10% w/v) of NSB1 and NSB2 were heated for 6 h at 70°C with continuous stirring. Then, the suspension was cooled for about 12 h at 20°C. The obtained spongy samples were washed with water followed by drying at 50°C for 24 h. It was then stored in an airtight container. The retrograded starch blends were coded as RSB1 and RSB2, respectively.^[7]

Fourier transform infrared spectroscopy (FTIR)

The FTIR spectrum of all samples was recorded using FTIR-8400 Spectrometer (Shimadzu, Japan) between a frequency range 400–4000 cm⁻¹. Dried KBr was mixed with starch blend samples for determination.^[8]

Amylose content analysis

The iodometric method was used to determine the amylose content in native and starch blends. Each starch blend was taken in a quantity of 100 mg for dispersion in 1 mL of (95% w/v) ethanol and was heated for 10 min on the water bath. The solution was then kept for cooling at room temperature and the volume was made up to 100 mL through Millipore water. A mixture of 1 mL 1N Acetic acid and 2 mL iodine was taken to mix with 5mL of the above solution and diluted up to 100 mL using distilled water.^[9] Absorbance of each samples was determined against the blank at the

wavelength of 620 nm in the Ultraviolet spectrophotometer (Shimadzu UV 2450, Japan).

$$\% \text{ Amylose} = 3.06 * \times \text{Absorbance} \times 20 \quad (1)$$

*3.06 is the conversion factor.

Water holding capacity

Water holding capacity was determined by suspending 1 g of starch blends in 15 mL of distilled water and stirred for 1 h. The resultant solution was transferred into a centrifuge tube. It was then centrifuged for 10 min at 3000 rpm (Remi R8C, India). Weighing of precipitated starch samples was performed after discarding the supernatant.^[10] Water holding capacity was determined by the following formula.

$$\text{WHC}(\%) = \left(\frac{W}{W_i} \right) \times 100 \quad (2)$$

Where W is the weight of dried polysaccharide.

Moisture content

1 g each of NSB1, NSB2, PSBA1, PSBA2, PSBA3, PSBB1, PSBB2, PSBB3, RSB1, and RSB2 samples were transferred on a clean Petri dish. It was then dried in hot air oven (Scientific Corporation India Ltd., India) for 2 h at 90°C until a constant weight was achieved. The calculation of moisture content was done as percentage loss in weight.^[11]

Solubility power and swelling power

Different starch blend (1% w/v) suspension was prepared and heated in a temperature-controlled water bath (Remi RSB 12, India) for 30 min at 30–90°C with constant agitation. The samples were subjected to centrifugation for 15 min at 3000 rpm (Remi R8 C, India). The supernatant was poured out and the adhered residue weighed (W_{ss}). It was dried (W_{su}) in a hot air oven (Scientific Corporation India Ltd, India) at 65°C for 24 h at constant weight.^[12] Solubility and swelling percentages were calculated using the equation,

$$\% \text{ Solubility} = \left(\frac{W_{su}}{W_i} \right) \times 100 \quad (3)$$

$$\% \text{ Swelling} = \left(\frac{W_{ss}}{W_i \times (100 - \% \text{ solubility})} \right) \times 100 \quad (4)$$

Micromeritics

Bulk and tapped densities

A 5 g quantity of native and thermally treated starch blends were placed in 10 mL measuring cylinder and the occupied

volume was noted as bulk volume. Then the measuring cylinder was tapped 100 times on a plane surface platform from a height of 1 inch at an interval of 2 s and then tapped volume measured.^[13] Bulk and tapped densities are calculated through following formulas

$$\text{Bulk density} = \frac{\text{Mass of Powder}}{\text{Bulk Volume}} \quad (5)$$

$$\text{Tapped density} = \frac{\text{Mass of powder}}{\text{Tapped volume}} \quad (6)$$

Percent compressibility index

Percent compressibility of the powder mix was calculated through Carr's index and Hausner's ratio using the following formulas.

$$\text{Carr's index} = \frac{\rho_{\text{tapped}} - \rho_{\text{bulk}}}{\rho_{\text{tapped}}} \times 100 \quad (7)$$

$$\text{Hausner's ratio} = \frac{\rho_{\text{tapped}}}{\rho_{\text{bulk}}}$$

Angle of repose

Angle of repose was performed using fixed funnel method. A funnel was clamped onto a stand. 10 g starch blend samples were weighed and gradually transferred through the funnel until peak of the heap touches tip of the funnel. The angle of repose was determined by measurement of height of starch sample heap (h) and the radius (r) was calculated by dividing mean diameter (d) of base the into half.^[14] Angle of repose for each sample was derived by employing the given formula:

$$\theta = \tan^{-1} \left(\frac{h}{r} \right) \quad (8)$$

Statistical analysis

All the data reported are an average of triplicate observations. The data were expressed as means \pm standard deviation.

RESULTS AND DISCUSSION

FTIR spectroscopy

All native and modified starch blends were assessed using FT-IR for the determination of the chemical changes due to modifications. The obtained data are graphically represented in Figures 1 and 2. A wide band at 3177 cm^{-1} in case of

all native and heat treated 1:1 starch blends and at 3110 cm^{-1} in case of all 2:1 blended starches might be attributed to -OH bond stretching.^[15] The 1:1 spectra showed two characteristics peaks near 949 cm^{-1} and 1150 cm^{-1} while the two characteristic peaks 1:2 were observed around 977 cm^{-1} and 1155 cm^{-1} , indicating the presence of -CO stretching.^[16] The bands near 1665 cm^{-1} for 1:1 blend and 1675 cm^{-1} for 1:2 blends were assigned to distortion vibrations of hydroxyl groups, while the peak near 2950 cm^{-1} for both 1:1 and 1:2 blended native and modified starches was ascribed to asymmetric stretching of -CH. Thus, it can be concluded that there were no chemical changes in both 1:1 and 1:2 blends due to thermal treatment.^[17]

Physicochemical properties

Amylose content

It is observed that amylose content is high for Pregelatinized blends of maize and potato starches in 1:1 (PSBA1, PSBA2, and PSBA3) and 1:2 (PSBB1, PSBB2, and PSBB3) as well as retrograded blends (RSB1 and RSB2) than their counterpart native starch blends. The amylose content [Table 1] increased from 11.32 \pm 0.06% to 20.31 \pm 0.20% in 1:1 starch blend and 17.87% \pm 0.04%–33.57% \pm 0.08% in 1:2 starch blends during pregelatinization whereas an increase from 11.32 \pm 0.06% to 13.51 \pm 0.18% and 17.87 \pm 0.04% to 21.54 \pm 0.09% for retrograded 1:1 starch blend and 1:2 starch blends, respectively, compared to their native blends of starch. The increase in amylose content can result in better starch drug complex indicating the potential use of the heat treated starch blends in colon specific drug delivery systems.^[2,13,18]

Water holding capacity

Water holding capacity [Table 1] of PSBA1- PSBA3 and PSBB1-PSBB3 (363.65 \pm 0.22%–689.77 \pm 0.54%) and retrograded blends RSB1 and RSB2 (679.37 \pm 0.57–732.27 \pm 0.34) was greater than that of native starch blends, NSB1 and NSB2 (218.73 \pm 0.13–325.48 \pm 0.68). It has been observed that the unfastened association of amylose and amylopectin molecules in the thermally treated starch blends is the main reason for high water holding capacity.^[19] Thus, this high water holding capacity of the starch blend can find its potential in formulation of hydrogels.^[20,21]

Moisture content

The moisture content is presented in Table 1 showed that all native and heat treated blends were in the range of 10.10 \pm 0.03%–15.42 \pm 0.03%. Hence, the moisture content of all the blends was found to be within the range specified by the Indian Pharmacopoeia. Thus, the low moisture content of the modified starch blends enables them to have better flow ability of granules during preparation of formulations.^[22,23]

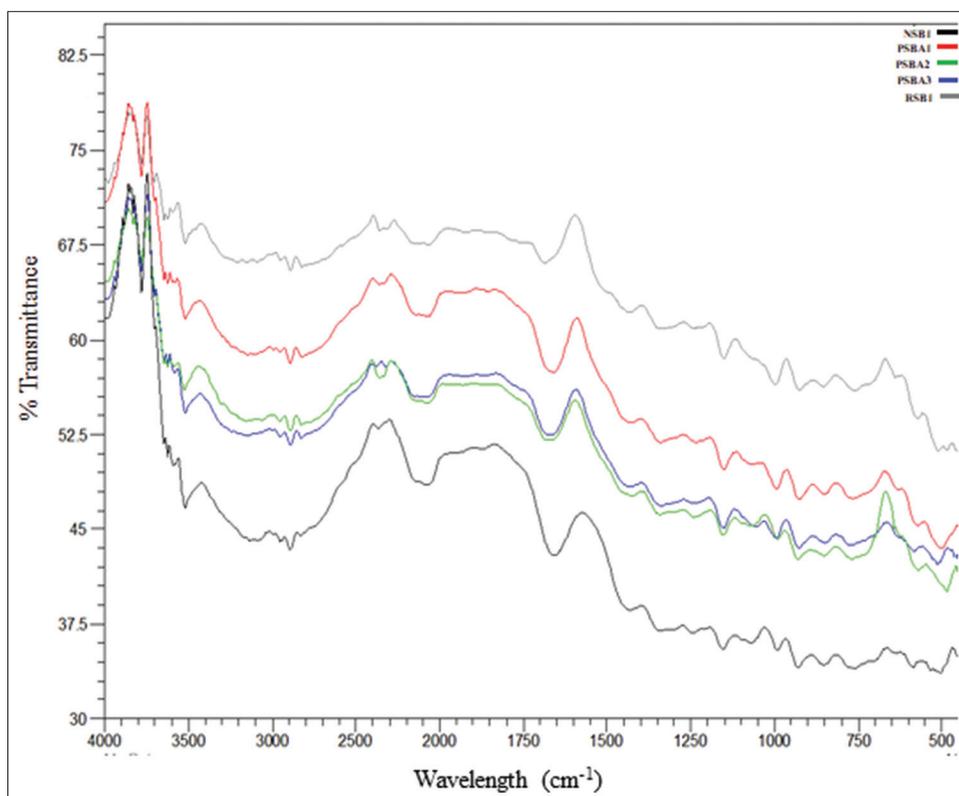


Figure 1: Fourier transform infrared spectroscopy spectrum of 1:1 starch blends

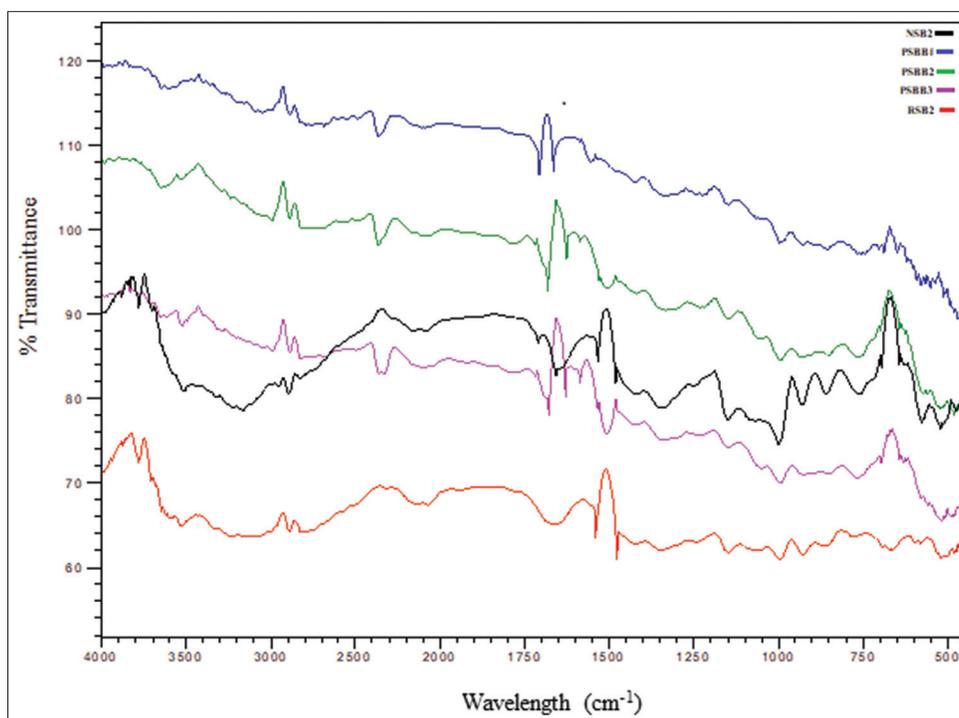


Figure 2: Fourier transform infrared spectroscopy spectrum of 1:2 starch blends

pH

The pH [Table 1] of all the starch blends was found to be weakly basic (7.15–7.46) but was within the range of British Pharmacopoeia specification for pH of heat

treated starch (4.50–7.50). Thus, the starch blend can find its potential application in extended release of pH insensitive drugs like Gliclazide for modulation of pH in the formulation.^[24]

Swelling and solubility

The swelling power and solubility of all starch blends at a temperature ranging between 30°C and 90°C are shown in Tables 2 and 3. The increase in swelling and solubility power is noticed with the increase in temperature of thermal treatment. Swelling power of starch blends varied from 3.22

± 0.52% to 16.75 ± 0.24% for native starch while it increases to 5.35 ± 0.25%–30.42 ± 0.41% for thermally treated starch blends. Solubility of starch blends varied from 7.31 ± 0.21% to 20.38 ± 0.32% for native starch while it increases to 11.11 ± 0.11%–34.42 ± 0.28% for thermally treated starch blends. It has been reported, swelling power is dependent upon amylose content, water holding capacity,^[25] and crystalline

Table 1: Physicochemical properties of native and modified starch blends

Sample	Amylose content (%)	WHC (%)	Moisture content (%)	pH
NSB1	11.32±0.06	218.73±0.13	10.10±0.03	7.21±0.06
PSBA1	14.17±0.55	363.65±0.22	13.21 ±0.03	7.25±0.11
PSBA2	16.31±0.23	464.78±0.07	14.60±0.04	7.15±0.29
PSBA3	20.31±0.20	493.73±0.13	15.30±0.13	7.16±0.16
RSB1	13.51 ± 0.18	679.37±0.57	13.20±0.44	7.25±0.04
NSB2	17.87±0.04	325.48±0.68	13.16 ±0.08	7.40±0.31
PSBB1	23.87±0.05	442 .02±0.25	13.78 ± 0.05	7.28±0.26
PSBB2	28.54±0.30	642.61±0.07	14.31±0.12	7.13±0.34
PSBB3	33.57±0.08	689.77±0.54	15.02±0.20	7.22±0.30
RSB2	21.54±0.09	732.27±0.341	15.42±0.03	7.34±0.44

Table 2: Swelling power of native and modified starch blends

Sample	Swelling power (%)						
	30°C	40°C	50°C	60°C	70°C	80°C	90°C
NSB1	3.34±0.35	5.57±0.18	7.89±0.82	10.12±0.22	12.28±0.18	14.45±0.25	16.75±0.24
PSBA1	6.27±0.45	8.53±0.22	11.22±0.41	13.13±0.29	15.36±0.17	17.57±0.43	19.82±0.22
PSBA2	6.53±0.42	9.86±0.35	13.24±0.22	16.33±0.28	19.40±0.23	21.40±0.42	23.78±0.40
PSBA3	7.52±0.41	10.26±0.45	14.31±0.22	17.90±0.23	20.14±0.12	23.42±0.22	24.42±0.19
RSB1	9.21±0.24	11.12±0.64	16.62±0.26	19.42±0.17	23.41±0.75	25.89±0.25	26.41±0.53
NSB2	3.22±0.52	4.79±0.78	6.74±0.28	8.23±0.24	10.32±0.45	12.43±0.52	14.02±0.42
PSBB1	5.35±0.25	7.96±0.73	9.12±0.72	11.41±0.23	14.31±0.45	18.22±0.42	19.04±0.84
PSBB2	7.51±0.19	10.13±0.41	14.68±0.42	16.74±0.28	19.43±0.43	22.42±0.45	24.72±0.12
PSBB3	9.31±0.20	12.55±0.41	16.48±0.33	19.33±0.25	21.22±0.42	24.13±0.14	29.90±0.23
RSB2	7.67±0.53	9.42±0.32	19.15±0.23	21.32±0.27	24.80±0.39	27.24±0.14	30.42± 0.41

Table 3: Solubility power of native and modified starch blends

Sample	% Solubility						
	30°C	40°C	50°C	60°C	70°C	80°C	90°C
NSB1	9.45±0.41	11.35±0.4	12.99±0.20	14.37±0.12	16.21±0.36	18.32±0.22	20.38±0.32
PSBA1	11.11±0.11	13.41 ±0.10	15.30±0.22	17.02±0.19	19.32±0.22	21.22 ± 0.35	22.96±0.21
PSBA2	12.50±0.15	14.11 ±0.22	17.82±0.34	19.21±0.15	21.22±0.25	23.25±0.34	25.42±0.61
PSBA3	15.12±0.15	17.55 ±0.19	19.80 ± 0.55	22.35± 0.38	24.63±0.53	27.31±0.25	29.40±0.32
RSB1	13.01 ±0.22	19.14 ±0.11	21.24±0.20	25.22±0.24	29.45±0.22	30.65±0.35	32.98±0.45
NSB2	7.31±0.21	9.33 ±0.35	10.90±0.11	12.80±0.55	14.10±0.26	16.32±0.25	17.30±0.47
PSBB1	12.70±0.62	14.33±0.33	16.47± 0.22	17.59±0.23	18.22±0.43	20.56±0.46	21.90±0.73
PSBB2	15.56±0.22	18.48±0.87	20.30±0.89	21.36±0.33	22.96±0.31	23.55±0.25	24.42±0.14
PSBB3	17.10 ±0.45	19.99±0.55	23.72±0.60	24.92±0.95	26.85±0.76	27.43±0.95	28.20±0.99
RSB2	12.35±0.22	17.90±0.12	19.52±0.52	23.76±0.42	25.81±0.23	30.18±0.32	34.42±0.28

Table 4: Powder characteristics of native and heat treated starch blends

Sample	Bulk density (g/cc)	Tapped density (g/cc)	Carr's index (%)	Hausner's ratio	Angle of repose (°)
NSB1	0.541±0.31	0.764±0.01	29.118±0.011	1.412±0.046	36.230± 0.014
PSBA1	0.671±0.21	0.840±1.01	20.119±1.011	1.251±0.100	21.431±0.12
PSBA2	0.648±0.02	0.806±0.03	19.603±0.341	1.243±1.010	20.153±1.11
PSBA3	0.686±0.20	0.831±0.08	17.449±0.304	1.211±0.641	16.130±0.20
RSB1	0.608±0.05	0.774±1.01	21.447±0.080	1.273±0.135	23.112±1.10
NSB2	0.643±0.04	0.822±0.02	26.776±0.241	1.278±0.002	33.330±1.52
PSBB1	0.535±0.07	0.673±0.05	20.505±0.244	1.257±0.005	25.653±0.37
PSBB2	0.550±0.14	0.679±0.01	18.999±1.001	1.234±0.014	25.541±0.60
PSBB3	0.608±0.04	0.738±0.012	17.615±1.11	1.21±0.007	24.571±1.32
RSB2	0.604±0.09	0.756±0.07	20.106±0.372	1.251±0.004	30.241±0.41

properties^[26] of starches. The interaction between starch chains within amorphous and crystalline domains is the result of the solubility and swelling power of starches. Thus, the enhanced swelling power of the thermally treated starch blend find its potential in gastroprotective delayed release drug delivery.^[27,28]

Micromeritic properties

The micromeritics properties shed light on the arrangement and packing of the particles and the compaction profile of a material. The Carr's index and angle of repose [Table 4] of thermally treated 1:1 blends were in the range of 17.44 ± 0.30%–21.447 ± 0.08% and 16.13 ± 0.20–23.112 ± 1.10 whereas thermally treated 1:2 blends were 17.61 ± 1.11%–20.50 ± 0.24% and 24.571 ± 1.32–30.241 ± 0.413, respectively, implying that the thermally modified starch blends have a good flow with fair compressibility unlike their unmodified counterparts with a very poor compressibility index between 26.776 ± 0.241% and 29.118 ± 0.011% and a passable angle of repose 33.330 ± 1.52–36.230 ± 0.01. Modification of the starch blends for the improvement of flow properties during process development will potentially lead to extended drug delivery.^[29,30]

CONCLUSION

The present work reveals a novel excipient with thermally treated blend of maize and potato starch in 1:1 and 1:2 proportions. The increase in physicochemical properties such as water holding capacity from 218.13 ± 0.13% to 732.27 ± 0.34% and amylose content (from 11.32 ± 0.06 to 28.54 ± 0.30) was noticed due to thermal treatment. The swelling and solubility were also found to increase due to pregelatinization and retrogradation indicating the modified have the potential to be used in gastroprotective delayed release drug delivery system. Enhancement of flow properties was observed for modified starch blends which is useful as an adjuvant for processing and formulation of solid oral dosage forms. In

conclusion, the modified starch blends of potato and maize showed promising synergistic effect as compared to the native blends as adjuvant in formulation of different drug delivery systems.

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