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Extraction of Starch from Different Sources: Their Modification and Evaluation of Properties as Pharmaceutical Excipient

Sraboni Kundu[1], Tanvir Muslim[1], Md. Azizur Rahman[1,*] and Md. Al-Amin Faisal[2]

[1] Department of Chemistry, Dhaka University, Dhaka-1000, Bangladesh

[2] Department of Biotechnology, BRAC University, Dhaka-1212

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Abstract

Starch from potato, maize and cassava was extracted using sodium metabisulphite solution. The percentage yield of starch obtained from all the sources was found to be quite satisfactory. The pregelatinized starch and carboxymethylated derivative of those starches were prepared. The pharmaceutical specification properties like solubility, pH, loss on drying (LOD), iron content, oxidizing substance, and microbial contaminations of the extracted starches, pregelatinised starches and carboxymethylated starches were evaluated as pharmaceutical excipient according to the British Pharmacopoeia. These finding conclusively suggest the feasibility of extraction and derivatisation of starch from potato, maize and cassava and their utilization as pharmaceutical excipient as well as in other commercial purposes.

I. Introduction

Bangladesh is an agricultural country. A plenty of food grains such as rice, wheat, maize etc. are cultivated here. These food grains have a great use in our diet because all of these are the main sources of carbohydrates as well as starch. Not only these food grains but a huge amount of potatoes are also grown in our country as a source of starch. The starch is used not only as our food but also in different industrial fields due to their multipurpose uses. To meet up these purposes, Bangladesh is importing a lot of starch specially potato, maize and cassava starch from foreign markets for our different industrial sectors. Bangladesh has all these sources in a sufficient amount for the extraction of commercial grade starch which may successively reduce this import and save the foreign currency. One of the major sections of utilization of native and modified starch is in the pharmaceutical industry. Some of the agro-based industries of our countries are manufacturing starch but this does not fulfill the specification of British Pharmacopoeia[1]. So the pharmaceutical sector cannot use these starches. Among the modified starches, pregelatinised and carboxymethyl starch are used in great extent for its binding capacity. Extraction and characterisation of starch from potatoes[2,3], rice[4] and corn[5,6] have been carried out. Corn starch and potato starches were acetylated[7]. Acetylation and evaluation of properties of rice starch has been investigated[8]. Carboxymethylation of potato starch and its characterisation has been performed[9,10]. Gelatinisation of potato starch and their characterisation have been carried out[11].

This is a report on extraction of starch from different sources, their physical (pregelatinisation) and chemical (carboxymethylation) modification and evaluation of their properties[1].

II. Experimental

General Methods

All solvents and chemicals used in the present work were analytical grade (E. Merck and BDH). All solvents were distilled before use. All drying were performed by freeze dryer and vacuum dryer. Fourier transform infrared (FTIR) spectra of the three extracted starches and the prepared carboxymethylated starches (CMS) were recorded using potassium bromide pellets by Shimadzu IR Prestige-21 spectrophotometer. pH-metric titration of CMS samples were carried out using Toledo MP-220 pH-meter for determination of degree of substitution. Different solutions and reagents were prepared using analytical procedure.

Materials and Methods

Collection of Samples: Potato (*Solanum tuberosum*) was collected from local market of Dhaka city. Two famous varities (BARI MAIZE-5 and SHUVRA) of maize (*Zea mays*) were collected from Bangladesh Agriculture Research Institute (BARI), Gazipur. The root of the cassava (*Manihot esculenta*) plant was collected from Vorosha Agro Chemicals, Gazipur.

Extraction of Starch

Starch from potatoes[2], maize[5] and cassava[2] was extracted separately using standard procedure. Potato and cassava starches were extracted using sodium metabisulphite solution[2]. Maize starch was extracted by steeping the maize with sulphur dioxide followed by degermination and gluten separation[5].

Characterization of starches

Test for characterization of the starches obtained from potato, maize and cassava were performed. The solubility, pH, oxidizing substances, iron content, loss on drying (LOD) and microbial contamination of the starches were carried out according to British Pharmacopoeia[1]. To

compare these results as pharmaceutical excipient, the standard specifications according to British Pharmacopoeia[1] are also presented.

Physical modification of starches

Pregelatinised starches were prepared from the extracted starches[12] and their different specifications as pharmaceutical excipient were studied.

Chemical modification of the starches

The extracted starches were separately carboxymethylated[13] and their degrees of substitution were determined by pH-metric titration[10,14,15]. The carboxymethylation of the potato, maize and cassava starches were carried out[13] using sodium hydroxide and monochloro acetic acid. The pharmaceutical specifications of carboxymethylated starches (CMS) were investigated.

Fourier transform infrared spectroscopic study of the extracted starches and prepared CMS samples:

Fourier transform infrared (FTIR) spectra of the three extracted starches and the prepared CMS samples were recorded.

III. Results and Discussion

Table-1 shows the percentage yield of potato, cassava and maize starches and were found to be 14.49%, 29.41% and 45.85%, respectively. The results are comparable to the standard values[1]. Not much difference was observed in appearance, aroma, solubility and microbial contamination of extracted starches, pregelatinised starches and carboxymethylated starches (CMS). Mostly the appearances of samples are white fine powder except for yellowish fine powder in extracted and pregelatinised starches of maize. Aroma of all samples is neutral. All extracted starches are partially soluble in cold water. On the other hand, all pregelatinised starches and CMS of starches are soluble in cold water and form gel. In all cases, the microbial contamination is less than 1000 CFU.

The pharmaceutical specifications of the starches such as pH, iron content, loss on drying (% LOD), and oxidising substance were determined (Table-2). These values were compared to those of standard specifications according to British Pharmacopeia[1] (Table-2). This revealed that the pharmaceutical specifications of the extracted starches are very similar to that of standard specifications indicating the feasibility of using these starches in pharmaceutical industries. The pregelatinised starches were prepared[12] separately from all the starches applying heat treatment. The percentage yield was found to be satisfactory (Table-1). The pharmaceutical specifications of these pregelatinised starches were determined (Table-3) and these values are comparable to the standard specifications[1]. The pharmaceutical specifications of the CMS of starches were determined (Table-3) and it appears that these values are very similar to that of standard specifications[1].

The percentage yield of CMS of starches from potato, maize and cassava were 38.51, 39.95 and 39.05, respectively (Table-1). The degrees of substitution of all the prepared CMS were determined by pH –metric titration[10,14,15]. pH vs. volume of sodium hydroxide titration curves were prepared and analyzed using software Curl Tipot (pH & acid base titration curve: Analysis and simulations) Version 3.3.1 (2008) for MS Excel. These results of degrees of substitution of all the CMS indicate that all three starches have been equally carboxymethylated (Table-1). Further, the CMS were characterized by Fourier transform infrared (FTIR) spectral study. FTIR spectra of the extracted starches and their CMS were recorded (Table-4). The typical infrared absorption frequencies of all extracted starch backbones and carboxymethyl group of all CMS were clearly observed. The FTIR spectra of starch[16] showed the broad absorption band at 3600-3200 cm^{-1} due to the stretching frequency of the –OH group. The band at 2900 cm^{-1} is due to the C–H stretching vibration. The band at 1300-1000 cm^{-1} is for C-O stretching. Again FTIR spectra of CMS showed a broad absorption band at 3600- 3200 is due to stretching vibration of –OH group and band at 2900 cm^{-1} attributes to C–H stretching frequency. The presence of a new and strong absorption bands at 1600-1590 cm^{-1} and at 1400 cm^{-1} confirms the presence of carboxylate (COO^{-}) group. The band of CMS at 1300-1000 cm^{-1} is due to different C–O stretching. All the starches and their CMS showed the same type of infrared absorption peaks. This FTIR spectral study clearly indicates that the starches have been carboxymethylated up to the desired degree.

Table. 1. Percentage yield of extracted starch, pregelatinised starch and carboxymethylated starches (CMS)

Samples	Percentage yield of			Degree of substitution of CMS
	Extracted starch	Pregelatinised starch	CMS	
Potato	14.49	73.16	38.51	0.67
Maize	45.85	75.93	39.95	0.53
Cassava	29.41	78.41	39.05	0.62

Table. 2. Pharmaceutical specifications of standard and extracted starches from potato, maize and cassava

Samples	Variables			
	pH	Iron content (ppm)	LOD (%)	Oxidised substance (ppm)
Potato	6.44	0.34	15.01	15.13
Maize	6.85	0.29	14.84	15.61
Cassava	6.62	0.42	14.66	15.42
Standard	5.0 – 8.0	Maximum 10	Maximum 20	Maximum 20

Table. 3. Pharmaceutical specifications of pregelatinised starches and CMS of starches (potato, maize and cassava)

Variables	Pregelatinised starches			CMS of starches		
	Potato	Maize	Cassava	Potato	Maize	Cassava
pH	5.44	5.85	5.62	6.01	5.95	6.25
Iron content (ppm)	0.32	0.31	0.39	0.35	0.26	0.31
LOD (%)	10.12	9.05	9.21	7.85	7.11	8.01
Oxidised substance (ppm)	16.27	16.65	16.55	N.D.	N.D.	N.D.

N.D. = Not detected

Table. 4. Comparative FTIR study of extracted starches and their CMS

Type of vibration	Extracted starch of potato, maize and cassava		CMS of potato, maize and cassava	
	Band (cm^{-1})	Characteristics	Band (cm^{-1})	Characteristics
–OH stretching	3600-3200	Strong (broad)	3600-3200	Strong (broad)
–C-H stretching	2900	Medium	2900	Medium
Assymetrical stretching of carboxylate ion ($–COO^-$ group)	—	—	1600-1590	Strong
Symmetrical stretching of carboxylate ion ($–COO^-$ group)	—	—	1400	Strong
–C–O stretching	1300-1000	Strong	1300-1000	Strong

IV. Conclusion

From this investigation, it can be concluded that starches such as potato, maize and cassava from native source can be extracted successfully. Their pregelatinisation and carboxymethylation can be carried out upto the desired degree of utilization. All these starches, pregelatinised starches and carboxymethylated starches are analysed and found to have the characteristics to fulfill pharmaceutical specifications according to British Pharmacopeia[1]. These observations, tests and analytical data reveal that these extracted starches and their derivatives may be used as pharmaceutical excipient as well as for other commercial purposes.

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