

A Brief Review on Thermo-rheological Properties of Starch Obtained from “*Metroxylon sagu*”

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Abstract: In the present era of diverging pharmaceuticals upcoming in the market, those from that of natural origin are gaining increasing importance. In this context, starch when obtained from natural source has high orientation to be used as pharmaceutical excipient. Among the natural sources, in the present review we have discussed about *Metroxylon sagu* i.e. sago starch as a potential source to be used as pharmaceutical excipient. We moreover have gone into details of sources, geographical area which can be exploited for further research work. We too have dealt with the properties like thickener, binding properties and various thermal and rheological properties. A section having brief discussion indicates the parameters affecting the grade and quantity of starch for use as excipient. The basic parameters we have included are like, amylose and amylopectin concentration, effect of salts, sugars, gelatinization and retrogradation which have a high impact upon grade of starch and its quality. Going through the review, the fact was observed that there are various parameters which can directly and indirectly affect starch grade and hence type and quality of formulation.

Key words: Sago starch • Thermo-rheological properties • Gelatinization • Excipient • Swelling behaviour

INTRODUCTION

About the Plant: Sago starch (*Metroxylon sagu*) is produced from the pith of the cycads of the genus, *Cycas*. *Cycas revoluta* is also referred to as sago palm, Japanese sago, or king sago and *Cycas rumphii* as tree sago or queen sago [1].

Sago initially is present in South East Asia and it could be produced from sago palm (*Metroxylon* spp.), which is also better known as rumbia. It is an important resource especially to the people in rural areas because it has various uses especially in the production of starch either as sago flour or sago pearl [2, 3]. Focusing, the geographical distribution, *M. sagu*, grows in freshwater swamps in Southeast Asia and many islands in the western Pacific. Amazingly, more than 200 kg of starch (sago) can be obtained from a single palm (Table 1). It has already been studied that ten or more of morphologically variable species of these were mutually correlated. Analysis of the various principal components revealed the fact that component analyses of the same

Table 1: General description of Plant

Kingdom	Plantae
(Unranked)	Angiosperms
(Unranked)	Monocots
(Unranked)	Commelinids
Order	Arecales
Family	Arecaceae
Genus	<i>Metroxylon</i>
Species	<i>M. sagu</i>
Botanical Name	<i>Metroxylon sagu</i> ,

morphological variables showed that neither armature (with presence or absence of spines) nor geographical separation was reflected clearly in the quantitative morphological variation of the plant. Similarity on the ground of genetic, quantitative, morphological, geographical and armature data has already been tested for correlation [4]. In spite of this fact; it has been reported in various literature that results only showed a significant correlation between genetic and geographical distances. The major conclusion drawn from the fact and

reported findings was that the geographical distribution defines some weak patterns in the genetic variation, whereas the genetic variation does not reflect any patterns in the morphological variation [5]. The manufacturing of edible starch products from sago palms of the genus *Metroxylon* is common to indigenous people throughout Melanesia but is unknown in places like Polynesia and even in areas where *Metroxylon* species are found widely. Therefore in accordance with the utility the use of *Metroxylon* as a starch source has also been reported in the isolated Polynesian island of Rotuma [6] and elsewhere where the capacity of plant *Metroxylon* has not been exploited. Some more species of *Metroxylon* like, *M. warburgii* (Heim) are gaining importance, since they have potent capacity to give starch of high grade and of potential use [7, 8].

Physicochemical Properties of Starch from Sago Palms (*M. Sagu*) at Different Growth Stages:

Different studies have been performed to determine grade, quality and properties of starch extruded, from different parts of the plants such as base and mid heights of the trunks of *M. sagu*. It is known that the morphology of starch though is same with all the species from various geographical sources taken into consideration but major difference lies in the amylose and amylopectin constitution. Moreover, particle size distribution, granule size and most important the pasting properties of starch have been found to vary from base to the top of bark from where starch exudates can be collected. The point to be considered is that, despite of various changes in chemical properties, the thermal properties of starch shows no variation [9].

This property is being exploited in the sense that same plant exudates can be collected together when it is to evaluate the thermal property and no significance change of chemical constituents is taken into account [10].

General Properties: Sago starch is one of the commonly used ingredients (e.g. thickener, stabilizer and gelling agent) in the food industry, the main use still remains as that of a thickener, stabilizer and gelling agents [11, 12]. In the present scenario, it has too been explored and exploited as a natural polymer which can serve as potential candidate for formulation of various dosage forms especially for those meant for oral administration.

Inclination of pharmacy sector towards the natural polymers for dosage form development has been a revolutionizing issue. Gelatinization and retrogradation are important physicochemical properties considered in food industry [13]. Sago starch has already been used in various fast dissolving preparations containing the active ingredient. This property has been reported in various literatures [14, 15, 16].

Gelatinized sago starch can also be stored for different times and under different temperature conditions for the investigation of retrogradation. Differential Scanning Colorimetry (DSC) was essentially used to monitor the thermal properties of retrograded starch. Increased extent of retrogradation (which is summarized by high melting enthalpy values and melting temperature) basically reduces enzyme susceptibility of sago starch and sago products at 37°C [17].

Carboxymethyl starch (CMS) with degree of substitution (DS) ranging from 0.1 to 0.32 was also prepared from sago (*M. sagu*) starch in non-aqueous medium using isopropanol as a solvent. The physicochemical, rheological and thermal properties of the starches were too investigated. At room temperature (25°C), carboxymethyl starch (CMS) hydrated readily, which resulted in higher swelling index as compared with native (unmodified) starch. Light microscopy revealed that CMS granules imbibed more water than native starch at room temperature and thus had caused a greater increase in granule size [12, 18]. Taking into consideration the specific viscosities and comparative studies they more so ever depend upon the content of amylose. Amylose content obtained from sago starch is reported to be higher than from those obtained from corn starch but quite similar to that of potato starch [12]. Complying various studies and research data it has been considered that moisture contents of the sago starch, its ash value, crude fat content, fiber content and crude protein content were in the limits which is typical for commercial application of starch (Table 2).

Table 2: Phytochemical properties of sago starch¹²

Properties	Values Range
Moisture content (%)	10.6 to 20.0
Ash value (%)	0.06 to 0.43
Crude fat content (%)	0.10 to 0.13
Fiber content (%)	0.26 to 0.32
Crude protein content	0.19 to 0.25

Table 3: Average values of sago starch and its components¹²

Part/ Component	Average values
Intrinsic viscosity (amylose)	310 to 460 ml/g
Molecular weight (amylose)	1.41x10 ⁶ to 2.23x10 ⁶
Molecular weight (amylopectin)	6.70x10 ⁶ to 9.23x10 ⁶
Gelatinization Temperature (sago starch)	69.4°C to 70.1°C

Under the average ambient temperature and humidity conditions the moisture content of most of the unmodified starches were found to range around 12%. Literatures support the fact that the total amylose contents (i.e. lipid free starch) in sago starches ranges between 24% and 31% compared to the apparent amylose content (starch with lipid) which is slightly lower than the reported range of 24-30% . A sight in the fact into the constituent especially amylose, it has been reported that the one can obtain from the fractionation procedure has a high iodine binding capacity of 19.5% while the other fraction as discussed before, amylopectin has very low iodine binding capacity of around 0.3% [11]. Depending upon the source and part of extraction it has been already analyzed that the starch extracted from lower part of trunk has lesser amylose content, as compared to starch obtained from large heights. Thus, it can be concluded that increase in height definitely abbreviates the content of amylose from the starch [12]. When compiled, the results of Scanning Electron Microscopy (SEM), has demarked that obtained starch consists of oval granules with an average diameter of around 30 μ m. Different characteristics of starch and related components as compiled in literatures have been tabulated as shown in the following Table 3:

The calculation of exponent 'a' in the Mark-Houwink equation and the exponent 'a' in the equation $R_g = kM^a$ have been found to be 0.80 and 0.58, respectively for amylose separated from sago starch and essentially when they are indicative of a random coil conformation. Concerning about the pasting properties, two types of pasting properties have been taken into consideration. The first is characterized by a maximum consistency immediately followed by sharp decrease in consistency while the second type is characterized by a plateau when the maximum consistency had reached [13, 17].

When we take more of the parameters into consideration, the effect of type of soil content has also been investigated. There exists a relationship between starch concentrations and activities of starch synthetic enzymes in sago palms (*M. sagu.*) which were obtained under acid sulfate and mineral soil conditions [18].

Plants that are grown naturally show that the growth in acidic sulfate soil is lower than that in mineral soil. Moreover lower amylose activity is observed in acidic sulfate soil than in mineral soil. These properties and regards are highly helpful since; lower amylose activity in mineral soil has compatibility to eliminate the degradation of starch, making the smaller granules suitable for storing large amounts of starch in a limited space inside cells, making it as its good potential, in this regard too [18]. Physical properties and biodegradability of the effect on biodegradability of the starches have already been studied in various literatures [19-21].

Modification of Sago Starch by Acid Treatment in Alcohol:

Generally, extracted starch is modified by using chemicals to overcome and improve the unstable properties of native/crude starch during processing. As with diagnosing the properties, it has been reported in various literatures that sago starch on treatment with hydrochloric acid in the presence of methanol, ethanol and 2-propanol at slight variation of temperatures between 40°C to 50°C for around 1 hour, the recovery has shown an extended yield of the modified starch (57%-94%) and the average granule size variation has been found to be within acceptable limits of 24.8 to 30.1 μ m. Thus, acid treatment has resulted in a pronounced effect on the degree of polymerization which has shown to progressively decrease with the decrease in number of carbon atom. Thus, sago starch can be modified through hydrolysis and alcoholysis [19, 23].

Hydrolysis patterns of five batches of sago starch was studied by using Novo Nordisk and Sigma α -amylases and glucoamylases which have reported the result that shows native sago starch was a poor substrate to the enzymes and the hydrolysis patterns were surface erosion, pitting and crevassing. After incubation with pH 3.5 acetate buffer at 60°C for 2 h, the hydrolysis pattern found was different: a single deep round hole developed regardless of the batch or enzyme(s) used. This step also has significantly reported an increased degree of hydrolysis. Granule size distribution results indicated that at about 67% hydrolysis, treated granule residues had the same mean size as native granules while untreated granule residues had two major size populations. Differential Scanning Colorimetry also suggested that amorphous regions of the untreated granule were preferentially hydrolyzed, however, upon pretreatment regions within the granule were more uniform towards enzymes action [19].

Swelling Behavior of Sago Starch: Swelling behavior of starch obtained from sago has been studied by Tester and Karkalas, which expressed the fact that the starch has a property to swell when it is heated in presence of water and hydrogen bonds which stabilize the structure of the double helices in crystallites are broken and replaced those of water.

Effect of Alkali on Swelling Behavior of Sago Starch:

It was investigated, that the alkali treated native starch and sago starch samples showed that swelling power of sago starch increases with time of alkaline treatment and it has been reported that sago starch treated with 0.5% NaOH had a higher swelling power and solubility than those of starch treated with 0.1% NaOH. Hoover reported that the extent of this alkali interaction is influenced by the amylose/amylopectin ratio and by the characteristics of amylose and amylopectin in terms of molecular weight/distribution, degree and length of branching and conformational studies [24, 25]. Studies have led to the fact that the swelling behavior of untreated starch is directly dependent upon property of the amylopectin content and wherein amylose acting as an inhibitor of swelling. Interestingly, quantity of sodium chloride also affects the swelling capacity and behavior of sago starch. Addition of sodium chloride to starch suspension has reported to show a critical behavior. It acts as swelling inhibitor as some protons of alcohol groups in the starch granule become exchanged by sodium ions. The swelling decreases with the increase of sago starch concentration [26].

Rheological Behavior of Sago Starch: Starch is one of the most important polysaccharides as discussed herein and as a raw material or as a food system in terms of nutritional qualities or functional properties. Starch is a biopolymer and can be defined as a homopolymer consisting only one monomer, D-glucose. In general, native starches are packed in granules. The characteristics of starch granular swelling, gelatinization and pasting are the important functionalities of the starch as an ingredient in food systems for controlling rheological properties of food as discussed earlier [27]. Rheology generally deals with the study of deformation and flow of matter. Deformation is the characteristics of solid material while flow to matter related to the liquid. In the solids elasticity directly shows its rheological property while for liquid, viscosity is the determining parameter. In context of food it can be said that they are neither viscous nor elastic but are rather viscoelastic.

The rheological response of any material is physically expressed as functions of either strain or strain rate or strain time. There are two types of stresses: those that act in a direction parallel to the material surface they deform, termed shear stress and those that acts in a direction perpendicular to surface of material they deform, called normal stresses [28].

Rheological behavior is basic consideration in food industry such as pasteurization, evaporation, drying and aseptic processing [27]. After the rheological study of sago starch at different temperature it was found that the rheological and viscosity of starch obtained from sago inversely related to the temperature. Rheological behavior of gelatinized sago starch solution was studied over the shear rate range of 13.61-704 s⁻¹ at various concentration and temperature ranges. A power law equation was used to describe the rheological behavior of the starch solution, while the effect of temperature can be evaluated by the Arrhenius equation [28]. The effect of starch concentration on apparent viscosity was studied using the exponential model describing the relationship between apparent viscosity and starch concentration. Consistency index ($\hat{\epsilon}$) increased with concentration and decreased with the increase of temperature. Flow behavior indices (ϵ) were within the range of 0.495-0.559 which indicated the pseudo plastic nature of gelatinized sago starch. The amount of starch and shear rate affect activation energy ($\hat{A}E$). Depending on the shear rate and concentration, activation energy is found to vary from 0.619 to 1.756 kcal mol⁻¹ [29]. A mathematical relationship correlating the various parameters (temperature, concentrations and shear rates) has been tested for its significance and validity [25, 26]. Rheological properties of starch paste and gels and other functional properties vary with species and variants. One of the most important features of starch in food systems is its ability to give structure by the formation of a gel. Dynamic viscoelastic methods have provided an excellent tool for studying rheological changes during heating and cooling without breaking the structure.

In various studies, starch was converted to glucose through two enzymatic steps: liquefaction and saccharification. But, when done with help of enzymes, starch was first converted by enzymatic liquefaction which yielded gelatinized starch and hydrolyzed to soluble dextrins, whereas, in the enzymatic saccharification, the dextrins were converted to glucose. In countries like Malaysia, although much work has been done for the conversion of sago starch to glucose, the data on the suitability of sago starch as a substrate for

glucose syrup production are still lacking. During the bioconversion, changes in viscosity and in reducing sugar concentration occurs which is an important fact to be taken under consideration [30].

Effect of Sugars on the Rheological Properties:

Different Studies have been carried out to investigate the effect of sugars on the rheological properties of sago starch. Presence of sugar also affects the swelling behavior of sago starch. The effects can be discussed in terms of the antiplasticizing effect of the sugars compared to water, the influence of sugar-starch interactions and also the effect of the sugars on water structure. It has already been reported that the storage modulus G' and the gel strength were significantly reduces in the presence of sugars. Generally G' decreases in the following order: control (water alone) > hexose > disaccharide > pentoses. It was showed that in the presence of hexoses the freeze-thaw stability of starch gels decreased while in the presence of disaccharides and pentoses the freeze-thaw stability was slightly improved [25].

Effect of Salts on Rheological Properties of Sago Starch:

The effects of various salts on the rheological properties of sago starch were studied using differential scanning calorimetry, small deformation oscillation and different deformation techniques as stated before. The presence of salts swelling properties, storage modulus G' , gel strength GS and gelation rate constants k , are affected and they now depend on the type and the concentration of salt. Their influence followed the Hofmeister series and the effect of anions seems to be more pronounced than that of cations. Sulfate ions increased G' , GS and k and finally reduced the swelling properties, whereas iodide and thiocyanate ions reduced G' , GS and k but increased the swelling properties [29]. Starch gelatinization in the presence of sugars has been described as a mechanical relaxation process affected by the mobility of aqueous sugar solutions [30]. Compounding such salts has great effects on the increase of gelatinization temperature of starch in fish starch mixture compared to sugar and MSG. It is also reported that the temperature range of gelatinization decreases with increase in fish content [31]. *Enzymatic hydrolysis of Sago starch:* Sigma-alpha amylase, glucoamylase and various other enzymes have been used to study biodegradation. All these enzymes fail to comply the limits of biodegradation after incubation at specified pH. Thus studies carried out suggest the use of particular temperature range and pH conditions for enzymatic hydrolysis of sago-starch [32-34].

Thermal Behavior of Sago Starch: In many studies, it was acknowledged that due to high water content, the amorphous regions of the granules imbibed water and swells, the finding results was found to be stripping or separation of starch chains from portions of these crystallites. When crystals when were treated at high moisture levels, they turned into crystallites, remaining to be melted at those levels. In the granules least stable crystallites were found to start to change first upon heating. Melting point is found to decrease, which results in quick melting of remaining crystallites. The whole process occurs quickly after absorption of water and is found to show a narrow or single DSC endotherm. It has been shown that at low water or moisture contents, cooperative melting does not takes place. This therefore results in a second endotherm, which represents crystallites melting after the cooperative process, at a higher temperature.

Effect of Sugars on the Thermal Properties of Sago Starch:

Different studies have been carried out to investigate the effect of sugars on the thermal properties of sago starch. Sugars were found to increase the gelatinization temperature T (gel) and gelatinization enthalpy; denoted as ΔH . In the presence of hexoses the freeze-thaw stability of starch gels decreases while in the presence of disaccharides and in case of pentoses the freeze-thaw stability was slightly improved [35].

Many investigations have tried to clarify the role and potential usefulness of hydrocolloids such as locust bean gum and guar gum to control rheology and modifying the texture of starch-based food products. Finally, it was concluded that the addition of hydrocolloids enhance or modify the gelatinization and retrogradation behavior of starch [35, 36]. The functions of adding hydrocolloid to starch, including the inhibition of retrogradation or the improvement of water-holding capacity for the starch system vary depending on the macromolecular characteristics of hydrocolloid phase behavior, which dominates the composite properties of starch/hydrocolloid system, which has been found to affect greatly not only by the mixing ratio but also by the molecular weight of each component [37].

Effect of Starch-hydrocolloid Mixture on the Thermal Behavior of Sago Starch:

Hydrocolloids are one of the important constituents used in food industry. They are used as thickening agents, stabilizing agents and texture modifiers and some others as detailed before.

They are found to act by mechanisms like, modifying the rheological properties of the aqueous phase between the dispersed particles. Researchers have already demonstrated that hydrocolloid have ability to form viscous solution at relatively low concentration and which that are only slightly affected by pH, added ions and/or heat processing. Several studies have been reported that the addition of hydrocolloids enhance or modify the gelatinization and retrogradation behavior of starch [36-39].

Effect of Starch-sugar Mixture on the Thermal Behavior of Sago Starch: Mobility of aqueous sugar solutions has ability to affect starch gelatinization. Many studied reported that the gelatinization temperature of starch increased and the swelling of starch granules decreases in the presence of sugars [37]. It was also studied that, generally, monosaccharides delay gelatinization less than disaccharides, except maltose [38].

Effect of Sago Starch-salts Mixture on Thermal Behavior of Sago Starch: Referring many literatures it can be quoted that salts can cause an elevation or depression of gelatinization temperature and gelatinization enthalpy, depending on the types of salt and their concentrations used [39]. It can also be concluded that salts like sodium chloride and Calcium chloride combination at low concentrations, slightly increased the T_p of sago starch but, when the concentrations increased further (up to ~2 M for sodium chloride and ~1 M for Calcium chloride), these temperatures decreased. It has been found that when sodium chloride concentration increased, the enthalpy value also consequently decreases. However, sodium chloride has been found to exhibit a maximum inhibitory effect on starch gelatinization at a concentration of between 6 to 9% [38, 40].

Use of Sago Starch in Some Food Production: Heat moisture treatment is a promising technique for improving the quality of sago noodle produced from sago starch and is performed by exposing the starch to high temperatures (110°C, 16 h) at moisture content of 25% [41]. Cross-linked sago starch has potential to be used for the partial substitution of wheat flour in the production of alkaline noodles. In market instant noodles, which have low levels (0.1-0.3%) of alkaline salts added as a texture improver are not classified as alkaline noodles because they lack the strong alkaline flavour and colour associated with the addition of high levels (0.5-1.5%) of alkaline salts.

It has been increasingly under consideration that production of white bread, which is consumed and rapidly rising in Indonesia is made of sago starch which can participate in percentage up 40% of the content of the white bread [42]. It has also been reported by various works in, Malaysia, that sago starch is widely used to produce 'tabaloi', which is one of the local biscuit delicacy as well as used in baking it into another traditional food called lempengs [43]. Sago starch is used in South East Asia in the production of many different types of foods such as vermicelli, bread, crackers and biscuits. Sago starch when mixed with water may be baked to form a product which is analogous to bread or a pancake [44-46]. As reported in literature, sago starch also serves as a low cost carbon source for production of various enzymes [8, 47, 48].

CONCLUSION

The above brief discussion aims to analyze the potential capacity of starch obtained from natural sources such as that of *Metroxylon* sp., commonly and commercially termed as sago starch. The literature compiles the fact of variation of type of starch obtained and the differing properties with difference with height of plant from which it is extracted. The portion stating properties deals with the physicochemical properties of starch and its comparison with those obtained with other natural sources such as potato. We can conclude the fact that various properties of starch such as thermal and rheological properties vary with differing temperature treatment and salt exposure. Also the obtained crude starch can be modified by treating it with alkali, alcohol and deriving its important constituents like amylose and amylopectin. Important starch derivatives are in use of industries and advantageous for the food market also. Overall, it can be concluded that simply sago starch or the modified sago starch possess immense potential to act as pharmaceutical excipient and has immense further scope to be worked upon.

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