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Acid Hydrolysed Starch for Drug Delivery System : A Review

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Abstract
Starch is a significant food product and a versatile biomaterial utilised in a variety of industries around the world, including food, health, textiles, chemicals, and engineering. The physicochemical features and usefulness of starch are largely responsible for its versatility in industrial applications. Starch has limited functioning and usefulness in its natural state. Starch is composed of amylose and amylopectin and deposited as granules of different sizes and shapes with semi-crystalline and amorphous concentric layers that show the 'maltese cross'. Starches from different sources show variable chemical composition as well as the structure of their components that are involved in thermal properties. Acid hydrolysis is a common chemical modification that alters the structural and functional characteristics of starch without causing granular morphology to be disrupted. Acid hydrolysis preferentially hydrolyzes amorphous areas, increasing the crystallinity and double helical content of acid hydrolyzed starch. The present state of knowledge about the effects of acid hydrolysis on starch structure and functionality is discussed in this review.
Keywords- Maltese cross, Amylose,Amylopectin,Amorphous

INTRODUCTION:

Starch or Amylum is a polymeric carbohydrate consisting of numerous glucose units joined by glycosidic bonds, which is chemically composed of two glucans polymers- Amylose and amylopectin. Each starch varies from one another based on their chemical composition- α-glucans , moisture , lipids , proteins and phosphorylated residues (1).

The polymers of glucose are glucans with different types of glycosidic linkages and anomeric configurations which are polysaccharides in the nature. There are mainly 3 types of these structural polysaccharides name α- Glucan , β-Glucan and mixed αβ- Glucan.(2)

Potatoes, corn, wheat, rice, tapioca, maize, and jackfruit are all good sources of starch. On complete hydrolysis, polysaccharides including starch, cellulose, and glycogen all generate d-glucose. D-glucose is widely accepted as the primary building component or basic unit of these higher complexes.(3)

Starch exists as partially crystalline granules in nature, which consists of two identical chemical components- Amylose Linear α(1,4) glycosidically linked and Amylopectin dendroidically branched, where α(1,6) glycosidic bonds forms the branching point. Different starches consists of different chains of polymers of Amylose and Amylopectin.(4).

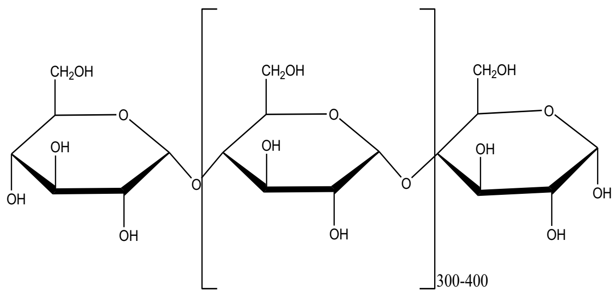

Fig a: Molecular structure of Amylose

AMYLOSE

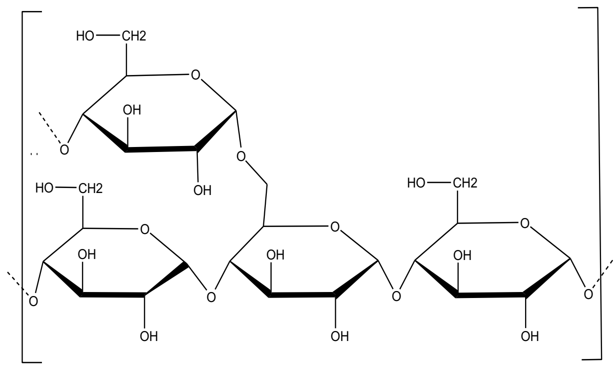

Fig b: Molecular structure of Amylopectin

AMYLOPECTIN

Starch is the major form of carbohydrate storage in green plants and is considered the second largest biomass, next to cellulose, produced on earth. Starch is a polymer that consists of six-member-ring glucose units (glucopyranose). The molecular weight of starch varies from 10^4 to 10^7 daltons(5)

PROPERTIES OF AMYLOSE:
PHYSICAL PROPERTIES:(6)

Table-1

Monomer unit name	Amylose
Density	1.25g/ml
Boiling point	627.7±55.0 °C at 760 mmHg
Bond Type	α glycosidic bonds
Chemical Formula	$(C_6H_{10}O_5)_n$
Odour	Unpleasant Odour
Appearance	White powder
Surface tension	74.4±5.0 dyne/cm
Solubility	Insoluble in water

ml(Milliliter),cm(Centimeter),g(Grams),mmHg(Millimetres of Mercury), °C(Degree Celcius)

CHEMICAL PROPERTIES:

1) After analyzing with High performance size exclusion chromatography, the amylose was found to be forming a complex distinctive blue colour with iodine.
2) Amylose forms hydrogen bonds with molecules, which may render the molecule that is less susceptible to enzymatic degradation.(7)

PROPERTIES OF AMYLOPECTIN:

PHYSICAL PROPERTIES:(8)

Table-2

Monomer unit name	Amylopectin
Density	1.25g/ml
Boiling point	1173ºC
Bond type	α glycosidic bonds
Chemical formula	$(C_6H_{10}O_5)_n$
Odour	Unpleasant odour
Appearance	Reddish brown
Surface tension	75.4±5.0 dyne/cm
Solubility	Soluble in water

ml(Milliliter),cm(Centimeter),g(Grams)

CHEMICAL PROPERTIES:(9)

Table-3

Chemical Formula	$[C_6H_{10}O_5]n$
Molar Mass Variable	Variable
Appearance	White Powder
Solubility in water	Soluble

All the starches are known as waxy starches because the endosperm tissue from which it is derived is waxy in appearance and these tissues contain a minimal amount of amylose in there granule composition(<15%).Waxy starch requires high energy for gelatinization due to its high crystallinity(10).

The physical properties of starch such as gelatinization, retrogradation-temperatures, and enthalpies is observed by the DSC (Differential Scanning Calorimetry) thermogram, where the gelatinization is shown as an endothermic peak located between 68ºC and 85ºC as gelatinization is an important starch property, because it indicates a change in its crystalline structure and functionality, and it improves the availability of starch for amylose hydrolysis. Whereas the Rapid Visco Analyzer(RVA) is used to observe the functional and structural properties of the starch such as- Water absorption capacity, Swelling index, Starch solubility, Rheological behavior (11).

STARCH PROPERTIES:

The functional properties of starch mainly include- Hydrolysis, Gelatinization, and in vitro digestion properties of starch. Insoluble starch granules when heated in water, the granules absorb water and swell this results in the disruption of crystalline structure which results in leaching of soluble starch in water which is known as gelatinization that is widely used in food industries.(12)

Based on its nutritional values starch can be divided into three categories- Rapidly digestible starch, slowly digestible starch and resistant starch. The resistant starch is not digested by the small but undergoes fermentation in the colon.(13)

In industrial purpose, the starch is used in coatings, sizing in paper, textiles, carpets also as binders, adhesives, absorbants, encapsulants, bone replacement implants, bone cements, drug delivery systems, tissue engineering scaffolds.(14)

The interaction of amylose and amylopectin with iodine is the most typical chemical characterisation. Amylose produces a bluish colour when it combines with iodine, whereas amylopectin produces a reddish brown colour. The discrepancy corresponds to the affinity that each molecule has for iodine , amylose binding on average 20 percent of its weight of iodine at 20oC whereas amylopectin binds less than 0.2 percent (w/w)(15)

The amylopectin component of starch determines its semi-crystalline structure.Water absorption causes amylopectin-amylose phase separation and crystallinity loss in starch granules, resulting in amylopectin-amylose phase separation and crystallinity loss. The depolymerization technique can also be used to solubilize starch utilising extra water at high temperatures (over 80°C).(16)

METHODS OF ACID HYDROLYSIS OF STARCH:

Starch modification is done by alteration of the physical and chemical characteristics of the native starch to improve its functional characteristics, this is achieved through derivatization like Esterification, Etherification,cross-linking,grafting of starch- Decomposition(acid or enzymatic hydrolysis and oxidation of starch) or by physical treatment of starch using heat or moisture.Similarly, chemical modification is the introduction of functional groups into the starch molecules.(17)

Modified starch is prepared by treating starch or starch granules by causing the starch to be partilly degraded, the purpose of this modification is to enhance the properties of starch in specific applications such as to improve the increase in water holding capacity ,heat resistant behavior,reinforce its binding , minimizing syngersies of starch and improved thinking .(18)

In Acid hydrolysis, the hydroxonium ion attacks the oxygen in the glycoydic bonds and later hydrolysyes the linkage, by exposing the starch to mineral acids such as H2SO4, HCl, HNO3 and H3PO4 at temperatures below the gelatinization temperature.(19)

The acid hydrolysis occurs in two stages, the first stage proceeds with faster rate due to the influence of the interaction of various factors such as amylose and lipid amylose complex content, granule size,presence of pores on granule surface, and the second hydrolysis stage occurs at slower rate due to the interaction of factors such as the amylopectin content, extent of distribution of the α(1->6) branches between amorphous and crystalline areas of amylopectin, and the double helices packing degree within the crystalline area.(20)

Hydrolysis of starch samples takes place in a hydrochloric acid solution at 50°C for 24 hours. In the reaction mixture, the starch and hydrochloric acid concentrations must be 32g/ml and 1.5 percent (w/w), respectively. To stop the process, the suspension was adjusted to pH 5.0 with 3% (w/w) sodium hydroxide after incubation. The suspension was rinsed multiple times with distilled water, vacuum filtered, and dried overnight at 40°C. The dried starch was pulverised and screened at a distance of 250 metres. The weight percentage of the produced starch residue to the weight of the beginning starch material was used to compute the starch yield.(21)

Hot acid treatment is process that is used to hydrolyse the starch.The starch stock (1 g/L) is made. Two distinct acids are used to give separate acid treatments, which are labelled correspondingly. Heating time, temperature, and acid content are all factors to consider.varies in a sequential manner The amount of sugar in the blood is determined by DNS stands for Domain Name System. Using distilled water and DNS, a blank is created. reagent. Then it is followed by the hot acid optimization process treatments.(22)

EFFECT OF ACID HYDROLYIS ON STARCH:

Acid treatment produced an increase in the rapidly digestible starch (RDS) (20.2%) and slowly digestible starch (29.7%), but a decrease in the resistant starch (36.3%) content, as compared with its native counterpart.(23)

This modification induces molecular degradation through the cleavage of α−1,4 and α−1,6 linkages of starch molecules . Except for some erosion on granule surface, the resultant acid-hydrolysed starches usually do not show extensive morphological changes when observed under Scanning Electron Microscopy(SEM).(24)

After a prolonged acid hydrolysis, the study showed the presence of amylose double-helical crystallites in the native mutant starches, which contributed to the resistance of enzymatic hydrolysis at 95–100 ◦C.(25)

That increase in amylose was attributed to faster rate of amylopectin depolymerization and liberation of more linear fragments increase in apparent amylose during acid hydrolysis of starch(26)

Amylose content, swelling power and solubility index of native starch and modified starch-(27)

Table-4

Acid Treatment	Amylose(%)	Swelling power(g/g)	Solubility Index
Native	17.4	14.0	7.8
0 h	17.0	15.1	10
8 h	16.8	1.35	77
16 h	15.9	0.97	81
24 h	14.7	0.88	82
48 h	9.6	0.82	83
72h	8.0	0.78	85

h(Hours)

A-type starches are thought to be more vulnerable to acid hydrolysis than B-type starches. due of the susceptibility of internal chains' branching locations (i.e., the clustered vs. scattered branching structure). Glucose helixes in A-type crystalline starch are densely packed, whereas glucose helixes in B-type crystalline starch are less densely packed.(28).

Increasing the hydrolysis temperature obviously shortens the time needed for the same degree of starch hydrolysis. The kinetics of hydrolysis for Hi-maize and Hylon VII starches are shown here. Since Hylon VII starch is of B-type pattern and has high amylose content of 65.10%, its amorphous zones should be fewer and its hydrolysis rates under the same conditions slower than those of normal-amylose starches(29)

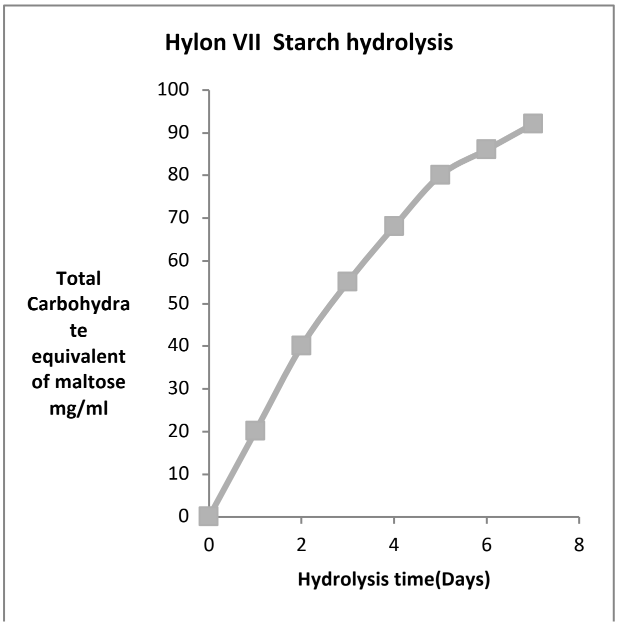

Figure c: Graph representing Total Carbohydrate content with respect to hydrolysis time for Hylon VII Starch

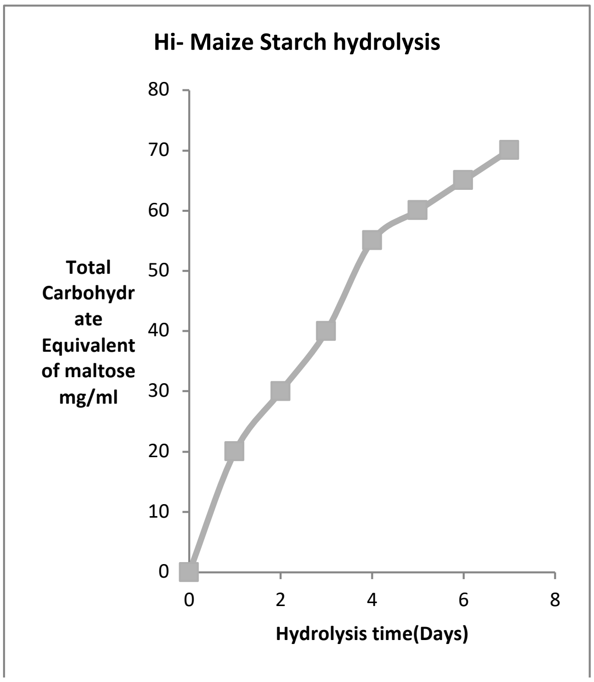

Figure d: Graph representing Total Carbohydrate content with respect to hydrolysis time for Hi-Maize Starch

The digestibility of starch is governed by the amount of resistant crystallites in organized regions and the amount of glycosidic linkages in the amorphous regions. The application of acid treatment had substantial effects on the outcome of autoclaving and β-amylolysis and, therefore, the digestibility of the final preparations has improved along with the decrease in particle size.(30) (31)

In acid hydrolysis, the hydroxonium ion ($H3O^+$) carries out an electrophilic attack on the oxygen atom of the α(1 → 4) glycosidic bond . In the next step, the electrons in one of the carbon–oxygen bonds move onto the oxygen atom to generate an unstable, high-energy carbocation intermediate . The carbocation intermediate is a Lewis acid, so it subsequently reacts with water , a Lewis base, leading to regeneration of a hydroxyl group (32)

The hydronium ion attacks the oxygen in the glycosidic bond during acid hydrolysis and then hydrolyzes the connection.Because the hydrogen bonds in amylose polymer chains are more exposed than in amylopectin polymer chains, this type of attack happens first.(33)

From the table we can conclude that the amylose content of starch decreases with increase in duration of acid treatment.(35)

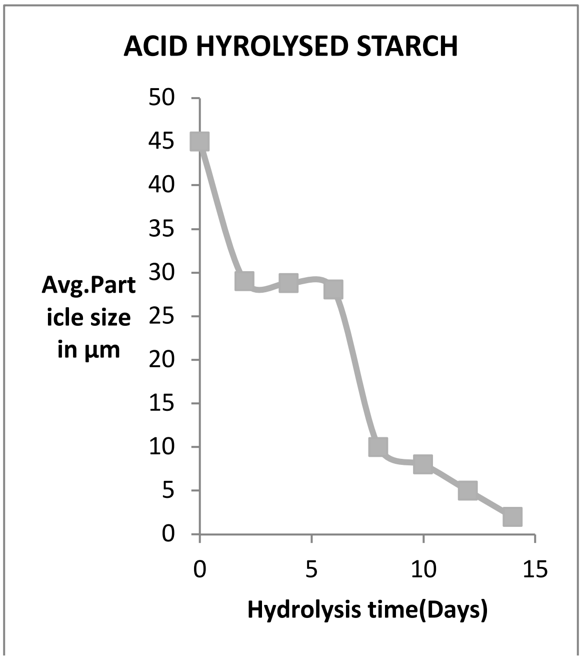

Figure e: Graph representing Average particle size with respect to Hydrolysis time for Acid hydrolyzed starch, µm (Micrometer)

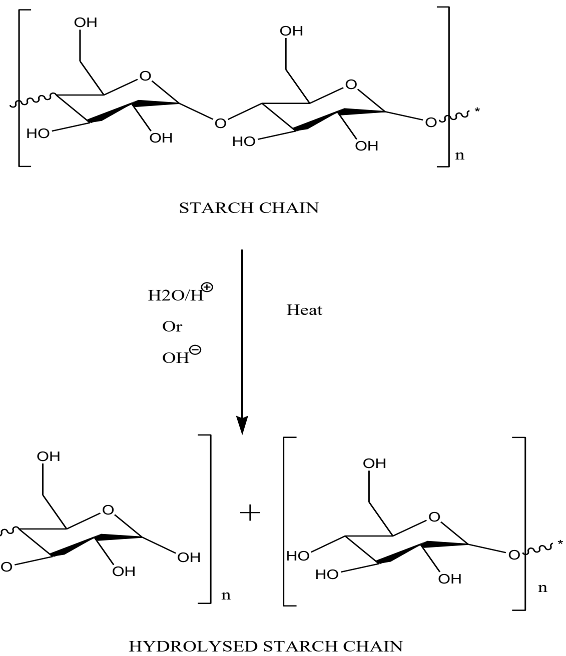

Figure f : Hydrolysis of α (1 →4) Glycosidic bond(34)

Table-5

Starch Type	Starch Pretreatment	Acid	pH	Temperature	Reaction time	Main Findings
Wheat Flour	NR	Conc. Sulphuric Acid	2-5	75-95ºC	15-105 min	Maximum Conversion (42%) of starch to reducing sugar obtained at 95ºC and pH 3
Potato Starch	NR	Dilute Sulphuric Acid,0-1%(v/v)	NR	70-150ºC	0-40min	Optimum conditions for glucose production obtained at 130ºC,1% acid and 7.5% solids loading for 30 mins
Waxy maize starch	NR	Hydrochloric acid 2.2N(or 2.2M)	NR	35ºC	3,8 and 15 days	Amorphous regions of starch granules are preferentially hydrolysed, increasing amount of rapidly digestible starch
Sago starch	NR	Hydrochloric acid,0.14M	NR	30-90ºC	6-24hr	Acid hydrolysis can decrease the gelling point and improve the solubility of sago starch in water
Sago starch	NR	Sulphuric acid,2.5%	1.0-2.0	121ºC	120min	6.6%(w/v) reducing sugar

NR- Not Reported, ºC(Degree Celcius), min(Minute),hr(Hour),M(Molar),N(Normal)w(Weight),v(Volume)

Applications of Acid Hydrolysed Starch:
The pharmaceutical tablets formulated which are acid modified showed higher tensile strength, lower friability, faster dissolution than the native starch and by increasing the acid hydrolysis time of starch was shown to found improved compatibility.(36)
The chemical acid hydrolysis of starch results in a product that is more resistant to food processing conditions (high temperatures, high shear, and low pH) and of higher viscosity compared to native, unmodified starch(37)
The acid hydrolysed starch is used as gelling agent, fat replacers/fat mimetic in low fat butter, as resistant starch-rich powder in slow digestible cookies, as adhesives to bond plasters and papers, as fillers in direct compression tablets, as raw materials in preparation of biodegradable films, as reinforcing agent in polymeric matrices to improve their mechanical and barrier properties.(38)
The most prevalent dissolving agent in tablet composition is starch. Tablet disintegrants are hydrophilic chemicals that expand in the gastrointestinal tract by absorbing water. Water absorption causes swelling, which causes the tablet to disintegrate into smaller fragments.(39)
Freshly made starch mucilage prepared to gel by acid treatment of the starch dispersion in water has been used extensively as binder in tablets and capsules production using the wet granulation technique. The starch mucilage is incorporated as a gel and functions as a glue to provide the necessary binding force that holds the powder particles together to form the required agglomerates. This also serves to ensure a uniform distribution and controllable release of the active pharmaceutical ingredient (API).(40)
Starch and its partially hydrolyzed derivatives (dextrins, maltose, and glucose) are good fermentation substrates for the bulk manufacture of a wide range of biotechnological products, including organic acids, antibiotics, vitamins, and hormones. Gluconic acid is produced by acid hydrolysis of strch by bacterial fermentation with Aspergillus niger or Acetobacter suboxidans (>97 percent yield). 103 The most effective salt is sodium gluconate, which is utilised as a chelating agent to make deltagluconolactone, which is used as an acidulant in food processing.(41)
The acid hydrolysis of drug improves the oral delivery of poorly water soluble drugs and increases the drug stability , it has been found that in biodegradable porous starch foam (BPSF), with a nanoporous structure has significantly improved its bioavailability after being hydrolysed by sulphuric acid.(42)

CONCLUSION:
The modifications are only possible due to the chemical reactivity of the constituent glucose monomers of the starch chains. Modified starches and their derivatives are used for various applications in the food industry The identification of novel starches with specific features and their potential for processing at large scales has piqued interest due to developments in the industrial sector.The lowest values of fractality are found in regular starch and starch treated with traditional acid hydrolysis, and after treatment, the value tends to stabilise at 4.0, indicating a rough surface due to acid assault, as expected. According to Table 5 Sago starch hydrolyses under sulphuric acid to produce proper Reducing sugars, which can be used for various application.

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Conflicts of Interest:
The authors declare that there are no conflicts of interest regarding this review.

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