



Pilot Plant Study for Investigation and Production of Liquid Glucose from Starch Slurry

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Abstract

In this research work, Pilot Plant study for the production of liquid glucose, a purified concentrated aqueous solution of nutritive saccharides obtained from maize starch has been investigated. As an agriculture dependent country, Bangladesh has a great opportunity to produce large scale liquid glucose from maize through cultivation of these plants at low cost. Among different processes, acid treatment process has been followed and the yield was satisfactory. Due to huge demand of liquid glucose in the local market for its wide applications in bakery, dairy, beverage, confectionery etc. a Pilot Plant Study has been carried out to assess the commercial and technical feasibility of the production process. Before Pilot Plant Study, process parameters were optimized in laboratory. According to the production process, required machineries were designed, fabricated and plant was erected. Dextrose Equivalent (DE) value of the obtained product was 45 which exist in between the range of standard specifications (40 -56). The produced products are characterized and characteristics are compared with that of the product available in the local market and Bangladesh Standard (BDS No: BDS CAC-9:2006). The obtained yield of the products is also analyzed. This study has great potential to produce liquid glucose in commercial scale.

Keywords: Pilot Plant Study, Liquid glucose, Starch, Acid conversion, Physical properties, Dextrose Equivalent

1. Introduction

Maize production in Bangladesh rises sharply in last decade and reached 5.4 million metric tonnes in Financial year 2022 – 2023 [1] is the main raw material for liquid glucose production. Among a variety of carbohydrates, starch is a naturally available polymer or polysaccharide formed by association of glucose monomers (Figure 1) through glycosidic bonds; therefore starch is a great source from which glucose, maltose, maltotriose and dextrin can be extracted by employing different methods [2, 3]. Glucose, also known as dextrose or sugar, is a crucial energy origin in living organisms as well as an indispensable constituent as a monomeric unit of a myriad carbohydrates such as oligosaccharides, polysaccharides.

In the arena of medical, pharmaceutical, food, brewing, textile industries and research sections, the products obtained by hydrolysis of starch (having glucose syrup, corn syrup and maltodextrin) has advanced and has been widely utilized. [4-7]. Through a variety of enzymatic treatment or chemical treatment, the conversion of starch to distinct sweeteners may be acquired. Many researchers reported the production of glucose syrup from different types of starch containing sources (such as maize, cassava and sorghum) and stated the advantages of enzymatic or chemical treatment. Yankovet al. [8] reported that enzymatic treatment is more beneficial to the latter because of presence of flavored breakdown materials and creation of unexpected colors. A. Zainabet al. [9] detained the lab scale production of liquid glucose by means of enzymatic hydrolysis of starch (millet, maize, and sorghum) and also noted that yellow maize and sorghum starch showed greater potential for liquid glucose production. Wang et. al. [10] reported enzymatic hydrolysis of sago starch and granule size distribution outcomes provided the 67% hydrolysis of starch. A lab scale study of production of maltodextrin and glucose syrup from banana starch by enzymatic hydrolysis was performed by L.A. Bello-Perez et. al. where maltodextrin equivalent was on the range of 7-11 [11]. Acid treatment of sago starch degradation in occurrence of raw starch degrading enzyme (RSDE) was also reported by M. Yetti et al. [12]. In many cases, enzymatic hydrolysis or treatment for industrial purposes are not efficient and also this process is very costly whereas acid treatment provides effective hydrolysis to produce maximum products.

On the other hand, ingredients for commercially manufactured foods like thickener, and sweetener for their moisture-retaining (humectant) features which keep foods moist and assist to restraint freshness, use of liquid glucose is in demand.

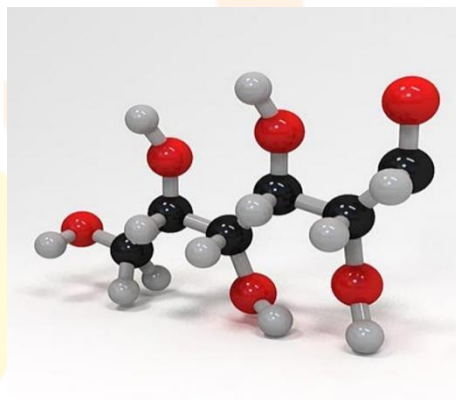


Figure 1: Structure of glucose molecule.

Keeping this consideration in mind about its huge demand in the food sector, an attempt has been employed to produce liquid glucose utilizing locally available ingredients and to investigate economic as well as commercial feasibility; a pilot plant study has been performed. Therefore the aims of this current research work was to design and fabricate a pilot plant for the production of liquid glucose for the study of process parameter for commercialization, other aims are to apply locally available technology to reduce fixed cost of the production process and subsequently to inspire the local small and mid-level entrepreneurs to set up industries for producing this product.

2. Materials and Equipment Design for Pilot Plant Study

The chemicals used in this study were of commercial grade, collected from local market and those were used without further purification. Sulfuric acid (H_2SO_4 , purity 99.99%, Molecular weight: 98.08, CAS Registration No: 7664-93-9, Merck, Germany) may be collected from the local market. Anhydrous Sodium Carbonate (Na_2CO_3 , purity $\geq 99.00\%$, Molecular weight: 105.99, CAS Registration No: 497-19-8, Merck, Germany) and activated Charcoal (Carbon (C), Molecular weight: 12.01, CAS Registration No: 7440-44-0, Sigma-Aldrich, USA) may be also collected from the local market.

After standardization of the process parameters, the units required for the production of liquid glucose has been designed, fabricated and installed in the Pilot Plant & Process Development Centre of BCSIR (Bangladesh Council of Scientific and Industrial Research). All of the fabrication materials, such as stainless steel sheet, pipes, valves and others required for fabrication of units were also collected from the local market. The fabricated units were a mixer, a demineralized water plant, an acid storage tank, a jacketed reactor, a neutralizer, (sodium carbonate) holding tank, a collection tank, a filtration unit and a vacuum concentrator.

2.1. Production Process

The production of liquid glucose has been carried out according to the following operational steps which are described sequentially.

2.1.1. Preparation of starch slurry:

To prepare starch slurry, measured amount of starch was charged in a clean mixing vessel. Demineralized water (DMW) was then added, 30 - 40% solid content of the starch slurry was maintained. Then sulfuric acid was added with continuous stirring of starch slurry to keep pH value within the range of 2.00 — 2.50 at RT (Room Temperature).

2.1.2. Reaction steps:

The obtained slurry of starch was then charged in a jacketed reaction vessel. Feed line and vacuum lines were closed and steam was allowed in the jacket to enhance temperature up to $140^\circ C$. Pressure of the in-line of jacket corresponds to the pressure of reaction vessel. Equivalent pressure against temperature $140^\circ C$ inside the vessel was about 2.0 Kg/cm^2 . Steam trap of the steam condensate line must be opened and therefore; steam line was closed after 30 minutes to maintain required temperature.

2.1.3. Neutralization:

The cooked slurry was neutralized in the reaction vessel by adding measured amount of sodium carbonate with gentle stirring.

2.1.4. De-colorization & filtration:

The neutralized slurry was slightly reddish in color and activated charcoal was used as a de-coloring agent as well as purifying agent in order to remove impurities. In this step, temperature was maintained at 70°C during addition of charcoal with gentle stirring. Neutralized slurry was allowed to cool to 50°C and drained out from the reaction vessel into a cleaned plastic drum. This black syrup mixed with charcoal was filtered to remove precipitated salt, coloring, impurities, carbon and other non-dissolvable impurities.

2.1.5. Vacuum concentration:

The obtained thick syrup was concentrated to 20% (by weight) in a vacuum evaporator at suitable temperature and pressure so that the final product remains transparent. Then the obtained product was collected in a food grade drum and ready to use.

The process for production of liquid glucose may be further cleared by the following flow chart.

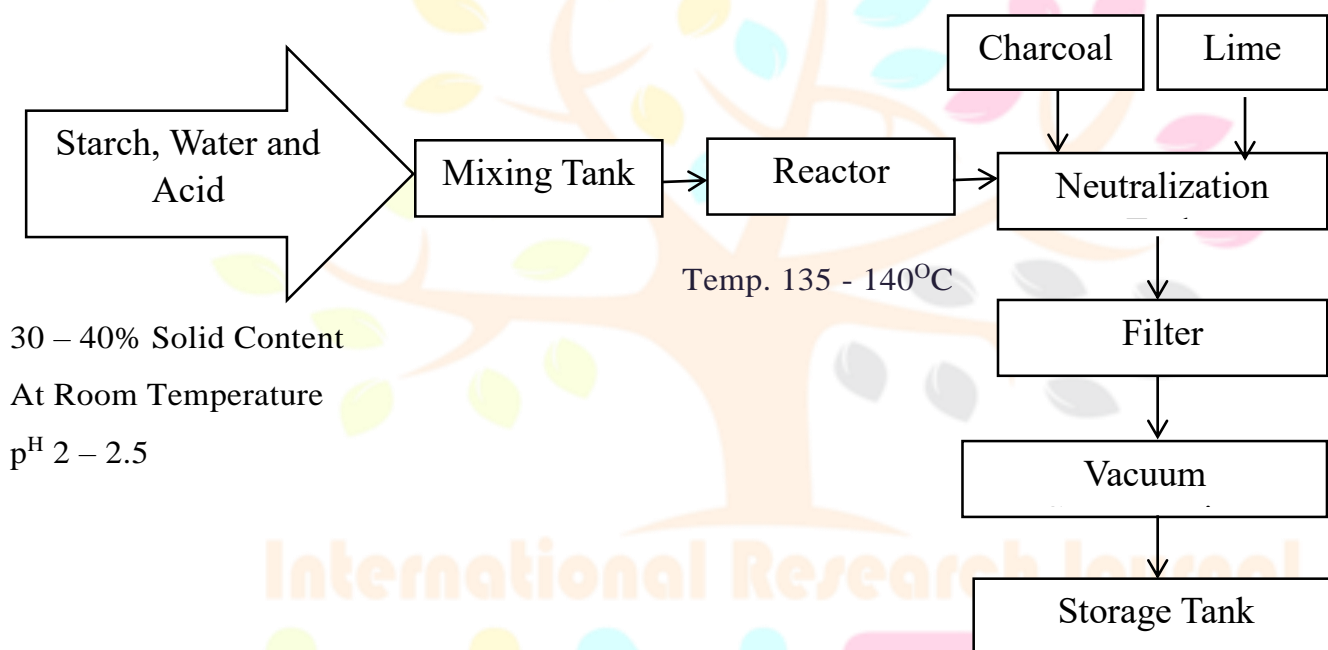


Figure 2: Flow chart of liquid glucose production in pilot plant.

3. Results and discussion

In this study, liquid glucose from corn starch has been produced in four different batches. Among which the most suitable and acceptable batch is third one. Dextrose Equivalent (DE) of the product obtained from this batch is 52. Table 1 shows the experimental results of several batches including the amount of charged starch (Kg), yield of product and dextrose equivalent. The first batch was started with 10 Kg starch which produced 7 kg yields having very poor sweet taste while the second batch produced 12.75 Kg yield from 15 kg starch having DE value 46 and sweet taste. In case of first batch, due to mechanical problem, expected temperature (135-140) was not achieved for required time period and therefore it was the main cause about slight conversion / unexpected conversion of starch to liquid glucose. As a result, DE value

was not obtained above 39. Enhancement of just only 5 kg starting material (starch), yield is increased than expected value (10.5 Kg) which indicates that this study is appropriate for pilot plant study for industrial purpose and this achievement of first and second batch is proved by further two batches delivering higher yields and standard DE value at the third batch. Although the last two studies showed 0.20 Kg difference at the similar conditions, the DE values are within the range of standard specification value, 40 – 56.

Table 1: The experimental results of several batches of liquid glucose production.

Batch Number	Amount of Starch (Kg)	Water (L)	Time (Hour)	Temperature (°C)	p ^H	Yield (Kg)	Taste of Product	Dextrose Equivalent
1	10.00	30.00	2	130	2.0-2.5	7.00	Poor sweet	Below 39.00
2	15.00	45.00	4	135-137	2.0-2.5	12.75	Sweet	46.00
3	20.00	60.00	4	135-140	2.0-2.5	17.40	Sweet	52.00
4	20.00	60.00	4	135-136	2.0-2.5	17.20	Sweet	45.00

3.1 Analysis of Raw Materials:

In this current study, pilot plant study has been carried out to produce glucose syrup from maize starch as raw material which is commercially available in large scale. The characterization of used starch has been carried out. A number of physio-chemical parameters of starch and Internationally Accepted Specifications IAS) are given in Table 2.

According to Table 2, collected starch was white in color and in powder form which are similar to IAS. The moisture (%) content (MC) of used starch is 10 which allowed by IAS range (9 – 12). Ash (%), protein (% dry basis) and fat (% dry basis) parameters of used starch also perfect according to IAS specifications (Table 2). p^H of used starch is 6.00 which is in between the range of 5.50–6.50, as the specified range of IAS.

Table 2: Internationally Accepted Specifications (IAS) and specifications of starch that is used for the production of liquid glucose in the Pilot Plant study.

Parameters	Internationally accepted specifications	Specifications of the used starch
Form	Powder	Powder
Color	Transparent White	Transparent White
Moisture (%)	9-12	10.00
Ash (%)	0.5 maximum	0.40
Protein (% dry basis)	0.4 maximum	0.40
Fat (% dry basis)	0.1 maximum	0.10
p ^H	5.50-6.50	6.00

3.2 Analysis of Liquid Glucose:

Liquid glucose that has been produced in pilot plant, been analyzed according to the standard specifications parameters and given in Table 3. Different physio-chemical parameters have been discussed below:

3.2.1 Total Solids:

25 ml of obtained sample was taken into a previously weighted beaker and inserted in a water-bath for 2 hours. The measured sample was then dried in an oven (Carbolite Gero Limited, UK; Laboratory oven PF) at 100°C for 3 hours. After that, it was cooled in a desiccator and weighted on a chemical balance. The result is shown in Table 3. Total solids % was evaluated by means of relation expressed below:

$$\% \text{ of total solids} = (\text{weight of dried sample}) / (\text{weight of initial sample}) * 100$$

3.2.2 Moisture Content (MC):

According to standard specifications, the accepted range of moisture content as 18.00- 22.50. Moisture content of the obtained product is 20 which proves that it follows the standard specification value. For moisture content determination, KERM (RH120-3) moisture analyzer was used.

3.2.3. pH:

100 ml syrup was taken in a 250 ml beaker and the pH was recorded by using calibrated pH meter (JENWAY, UK; 3510 pH meter). The pH of the obtained product was 5.00 which is similar to Standard specifications (Table 3).

3.2.4 Ash Content:

10 gm sample was weighted and taken into a porcelain crucible which was previously ignited and weighted. The crucible with sample was inserted in the muffle furnace (Nabertherm, GmbH; Germany) and maintained at 600°C and after heating it was allowed to cool in a desiccator and then the residue substance was weighted immediately. Therefore, % of ash content was calculated and evaluated by means of following relation and recorded in Table 3.

$$\% \text{ Ash} = (\text{Weight of crucible with ash} - \text{Weight of empty crucible}) / (\text{Sample Weight})$$

3.2.5 Refractive Index and Viscosity:

The obtained result of refractive index is 1.40 which is very close to standard specification value and BELLINGHAM & STANLEY LTD. (LONDON) was used for refractive index determination. Figure 2 shows the final product in this current study. Lide and David [13] reported the refractive index 1.4394 at 589.28 nm wavelength 60% glucose solution and provided close harmony with our study. Weight per cubic foot value of obtained liquid glucose is 88.37, which is similar with standard specification value (Table 3). The viscosity

(HAAKE, VT550) of liquid glucose is 29,600 (in centipoise unit) which does not show much difference from that of standard specifications (29,500).

Table 3: The standard specifications of a typical liquid glucose (higher sugar content) and that of the Pilot Plant product

Parameters	Standard specifications	Analytical results
Dextrose Equivalent (DE value)	40-56	52
Total solids (%)	78-81	80
Moisture Content (%)	18.00-22.50	20.00
pH (50% solution)	5.00	5.00
Ash (%)	0.30	0.30
Refractive Index (45° C)	1.4931	1.40
Weight per cubic foot (38°C)	88.37	88.37
Viscosity (Centipoise, 38°C)	29,500	29,600

3.2.6 Dextrose Equivalent (DE):

Due to precipitation (retro gradation) of the longest chain linear polymers, acid syrups including a Dextrose Equivalent (DE) value under 30 will tend to cloud upon standing condition. Through acid hydrolysis higher DE values like 80 or above 80 may be achieved, the extreme conditions needed to achieve DE values greater than 55. In that case, it enhances the formation of excessive amounts of glucose degradation which is difficult to remove during refining.

As a result, it causes yellowish color of the product. Very common and most suitable acid glucose product has a DE value of about 40-56, because of these upper and lower conversion limitations [14]. M.D.W. Samaranayakeer et al. [15] reported that high rate of continuous mechanical agitation in pilot plant study may cause the degradation of polymeric chain of the produced starch molecules and results in the production of more simple sugars. These obtained reducing sugars caused to enhance DE and Brix value in medium [16] range. From this study, obtained analytical result of Dextrose Equivalent (DE) value of products from second, third and fourth batches are 46, 52 and 45 respectively. Which are within the range of standard specification value 40 - 56.

4. Conclusion:

This current study developed a very easy and applicable process of liquid glucose production from corn starch through pilot plant study. Yield of liquid glucose is 86% from charged raw starch and DE value is 45 - 52; these achievements are strong indication for the production of industrial scale. The obtained liquid glucose is more viscous and followed different specification values. Its refractive index is 1.40, pH is 5.00 and moisture content exists within the accepted range. It can be utilized for various purposes like confectioneries, beverages and bakery. This process is very much effective not only for Bangladesh perspective but also for any other country.

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