Vol. 24, No. 2(2025) Bio25511
Revista Mexicana de Ingeniería Química

Construction of a hydrolysis reactor to obtain monosaccharides from a lignocellulosic residue

Construcción de un reactor de hidrólisis para obtener monosacáridos de un residuo lignocelulósico

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Received: January 22, 2025; Accepted: March 21, 2025

Abstract

The present work shows the design prototype of a pilot scale full mixing reactor made of stainless steel and which was tested in a dilute acid hydrolysis process. Computational Fluid Dynamics (LS Dyna software) was used to study the geometric configuration and hydrodynamic profile in preliminary tests (at a temperature of 120°C) to obtain reducing sugars from a lignocellulosic waste (pineapple waste), obtaining a maximum production of 45.5 g of sugars per 100 g of residual material at a time of 140 min. The concentration results obtained in the kinetics were adjusted to the Saeman kinetic model, obtaining a production constant k_1 of 0.0168 min^{-1} and a degradation constant k_2 equal to 0.0028 min^{-1} which showed that the production was greater than the degradation. of the sugars obtained.

Keywords: chemical reactor, design, Dyna software, kinetics, hydrolysis.

Resumen

En el presente trabajo se muestra el prototipo de diseño de un reactor de mezcla completa escala piloto fabricado en acero inoxidable y que fue probado en un proceso de hidrólisis ácida diluida. Se utilizó Dinámica de Fluidos Computacional (LS Dyna software) para estudiar la configuración geométrica y perfil hidrodinámico en pruebas preliminares (a una temperatura de 120°C) para la obtención de azúcares reductores a partir de un residuo lignocelulósico (residuo de piña), obteniéndose una producción máxima de 45.5 g de azúcares por cada 100 g de material residual a un tiempo de 140 min. Los resultados de concentración obtenidos en la cinética fueron ajustados al modelo cinético de Saeman, obteniéndose una constante de producción k_1 de 0.0168 min^{-1} y una constante de degradación k_2 igual a 0.0028 min^{-1} lo cual mostro que fue mayor la producción sobre la degradación de los azúcares obtenidos.

Palabras clave: reactor químico, diseño, Dyna software, cinética, hidrólisis.

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https://doi.org/10.24275/rmiq/Bio25511
ISSN:1665-2738, *issn-e*: 2395-8472

1 Introduction

Acid hydrolysis is a widely used chemical treatment to revalorize lignocellulosic biomass into valuable products. The main focus of acid hydrolysis is to increase the total surface area of the material, partially removing lignin external layer, reducing crystallinity index and polymerization degree to purify cellulose biopolymer (Rezania et al., 2020). From this, in literature it has reported either diluted or concentrated acid hydrolysis, and their employment depends on the physicochemical changes target on raw biomass. Concentrated acid hydrolysis is also known as swelling or de-crystallization process which aims on disrupting the recalcitrant nature of lignocellulosic matrix, solubilizing hemicellulose and cellulose fractions and breaking down lignin structure. Usually, this process performs at high acid loadings; >25 %w, moderate temperatures; < 60 °C and high solid-liquid ratios; > 1:2.5 (Wolfaardt *et al.*, 2021), however, due to the concentrated acid expensive reactors and resistant materials are needed, and thus, this becomes un-practicability and un-sustainability for large scale application.

Diluted acid hydrolysis is most preferable due to low acid loading and lower toxicity compared to concentrated acid, this process usually performs at high temperatures coupled to low acid concentrations and long reaction times and aimed to transform hemicellulose and amorphous cellulose fraction into monomeric sugars that serves as carbon source for the subsequent fermentation stages. The main disadvantage of this hydrolysis treatment is the low lignin removal (only acid-soluble lignin) and the formation of aldehydes like furfural and 5-Hydroxi methyl furfural (HMF), which are inhibitory for microorganisms growing (Gómora-Hernández *et al.*, 2020). In several papers, acid hydrolysis has been considered as critical step during lignocellulose revalorization, and thus, most of them have focused on operational conditions optimization from a wide range of biomass, the employment of either organic or inorganic acids as wells as the use of acid hydrolysis treatment as preliminary step for saccharification-fermentation processes to biofuels and bio-based materials production (Selvakumar et al., 2022; Solarte-Toro *et al.*, 2019). Despite the interest of this technology in the last decades, there is only a few of papers reporting scaling up strategies for improve acid hydrolysis toward large scale application. As any chemical process, scaling up is a challenge stage, since it depends on several techno-economic and chemical factors. Due to the heterogeneous nature of acid hydrolysis of lignocelluloses, the scaling up of a hydrolysis batch reactor involves not only chemical features but also mass-transfer limitations which could dominate the overall process; however, some authors suggest that using particles lesser than 840 μm diffusion contributions could be avoided (Gómora-Hernández *et al.*, 2022). Mass transfer depends on different factors like the physical characteristics of biomass; density, tortuosity, porosity, mechanical resistance, particles volume as well as the nature of acid solution; viscosity, density, concentration, protons diffusion and among others (Chen *et al.*, 2015).

Since batch reactor serves as homogeneous stirring tank, the geometrical considerations reported by Mc Cabe *et al.* (2007) can be used for its design. These considerations are not limited to stirring rod, deflectors and reactor body (height and width). In addition to geometrical reactor design, modelling by computational tools is an interesting alternative to depict processes behaviour before its real operation. The understanding of the behaviour of any process prior its large-scale implementation allows eliminating operational stages, reducing costs and thus, the cost-benefit balance and the performance are better.

Computational Fluid Dynamics (CFD) is a novel computational tool based on finite-element method commonly employed to solve differential equations and provides numerical approximation of physics phenomena (Zawawi *et al.*, 2018). For fluid dynamics, CFD performs dynamic simulations through 3D structures, which provides flow distribution, hydrodynamic – thermal profiles, velocity and pressure gradients and the hydrodynamic behaviour inside container (Llano-Serna *et al.*, 2019). Recently, this powerful tool has been used in a wide range of applications including electromagnetism, heat and mass transfer (Sabliov *et al.*, 2006), vehicles design (Sarkar *et al.*, 2019) and chemical reactions engineering (Khanghah and Jafari, 2022).

In this work, geometrical configuration design of a hydrolysis batch reactor, hydrodynamic profiles given by CFD analysis as well as the preliminary hydrolysis tests for sugar extraction from a lignocellulose source is reported and discussed. The main focus of the paper is reporting the geometrical design and construction of batch reactor on a homogeneous stirred-tank basis configuration and analysing by finite element method the hydrodynamics profile for proper fluid distribution through 3D structure. Preliminary hydrolysis assay and the kinetic behaviour for sugars extraction was also analysed and compared with some data reported in literature before.

2 Materials and methods

2.1 *Raw material*

Pineapple crowns were selected as model raw material to evaluate sugar extraction in a batch hydrolysis reactor. They were obtained from local markets and transported to Technological Institute of Toluca for further conditioning and study. Pineapple crowns were washed with distilled water to remove impurities like ground, sand and plastics sliced into small pieces of around 3 cm^2 and dried at 55 °C for 72 hours. After that, they were pulverized and meshed, and the particles lesser than 250 μm were recovered, characterized and used for hydrolysis assays. The content of holocellulose, lignin, moisture and extractives were determined according to a previous paper (Gómora-Hernández *et al*., 2020).

2.2 *Geometrical configuration of hydrolysis batch reactor*

Geometrical configuration of batch reactor was based on the similar mathematical relationships (Figure 1) reported by Mc Cabe (2007), which allow estimating the width of deflectors, dimensions of blades and propels and the width of batch reactor. For geometrical design, a total volume of 2 L was considered as calculus basis. It is important to note, that the geometrical dimensions are limited to a homogeneous stirred tank, so, other limitations like mass transfer and chemical factors are avoided. The consideration proposed in this paper is based on the nature of sugar extraction reaction during acid hydrolysis which has been considered as pseudo-homogenous reaction when solid particles are lesser than 840 μm, i.e. chemical reaction is carried out in homogeneous liquid phase (Arslan *et al*., 2012).

2.3 *CFD analysis of batch reactor and assembly*

The second part of hydrolysis batch reactor construction was the instrumentation and validation of stirring profile. For this, each component of batch reactor and a "U" thermal resistance was designed and draw employing CAD software. After that, individual drawings were export to V971_R8 LS Dyna Software, which was used as CFD tool to determine flow profile through batch reactor. Reactor body, stirring rod and thermal resistance were discretized by 1 mm hexahedrons. Since reactor configuration considers a headspace of around 30 % of the total height, air and liquid solution layers were considered for stirring profile.

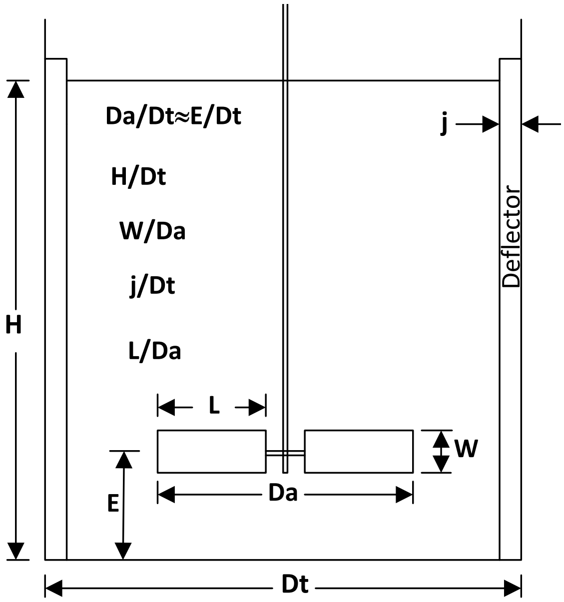

Figure 1. Geometrical configuration of homogeneous stirred tank.

Prior simulation, physicochemical parameters for liquid phase like density and viscosity as well as heat capacities of air and water was feed to the software. Density and viscosity of the mixed was determined by using a conventional glass densitometer and Ostwald viscometer, respectively. The mixed was comprised by 1 g of milled and dried pineapple crown with 20 mL of acid solution; this solid-liquid ratio was proposed based on a previous report (Gutiérrez-López *et al*., 2022).

After geometrical design and CFD study, steel-based reactor was constructed. The body and the down of the reactor were comprised by an only steel piece, while a flange was used as cover. Batch reactor was placed on a metal base and conditioning with mechanical and electronic systems. Stirring rod was connected to the mechanical rotor through bearing system, while thermal components were coupled to an electronic device; electronic control, thermal resistance and temperature dipstick, which maintains operational temperature within the desired range. Finally, reactor body was cover with glass wool to avoid thermal losses. To validate heating system, reactor was charged with distilled water and heating until up 100 °C. Samples were taken from purge valve at different times; temperature was measured using conventional mercury thermometer and compared to that registered by electronic device. Temperature was raised until 120 °C to verify the absence of steam leaks between reactor body and its cover and over mobile parts. Since hydrolysis reaction could be performed at long times, a pigtail pressure gauge was installed to monitor pressure changes attributed to high temperature. Pre-operational tests were performed to validate heating rates, fluid flow inside reactor and heat losses.

2.4 *Preliminary acid hydrolysis assays*

Acid hydrolysis of pineapple crown particles was carried out by triplicate at 110 °C using a solid-liquid ratio of 1:20. Sulphuric acid solution (1 M) was used as catalyst. Reaction was performed at a constant stirring of 250 rpm and the liquid samples were taken every 20 minutes until 240 minutes. Each sample was air cooled, centrifuged to eliminate suspended solids and the total amount of reducing sugars was quantified by Dinitro salicylic acid method (DNS) as was reported previously (Gómora-Hernández *et al.* 2022).

Kinetics for sugar extraction on batch reactor was studied by Saeman model. This model has extensively been used for acid hydrolysis of lignocelluloses and considers that hydrolysis acts as pseudo-homogeneous first order reaction in which chemical interaction performs in the homogeneous liquid phase, so, Saeman model does not consider limitations due to mass transfer and diffusion through solid porous material (Abril-González *et al.*, 2023; Arslan *et al.*, 2012). This model considers that acid hydrolysis involves 2 sequential first order reactions; the first related to monomeric sugars production from hemicellulose and amorphous fraction of cellulose, while the second is associated to thermal decomposition of the extracted saccharides into furfural (For C5 sugars) and HMF (For C6 sugars) (Ebikade *et al.*, 2018). The mathematical expression of Saeman model is shown in Eq. 1.

$$M = \frac{P_o\, k_1}{k_2 - k_1}\left[e^{-k_1\, t} - e^{-k_2\, t}\right] \tag{1}$$

Where M and P_o are the amount of reducing sugars and holocellulose, respectively, both given in g/ 100 g of raw pineapple crown, k_1 and k_2 are the kinetic constants (min^{-1}) for production and decomposition of sugars, respectively and t the reaction time in minutes. Sugar production yield was calculated according to the following equation:

$$M = \frac{v * C}{1000} * 100 \tag{2}$$

Where v is the volume of hydrolysate recovered after acid hydrolysis, C the concentration of sugars in g/L, 1000 a conversion factor for volume and 100 the calculus basis for mass. In addition, the maximum sugar production and the optimum time at which this production is yielded were determined employing the following equations:

$$Sug_{\max} = P_o\left[\frac{k_2}{k_1}\right]^{\frac{k_2}{k_1-k_2}} \tag{3}$$

$$t_{opt} = \frac{[\ln(k_2) - \ln(k_1)]}{k_2 - k_1} \tag{4}$$

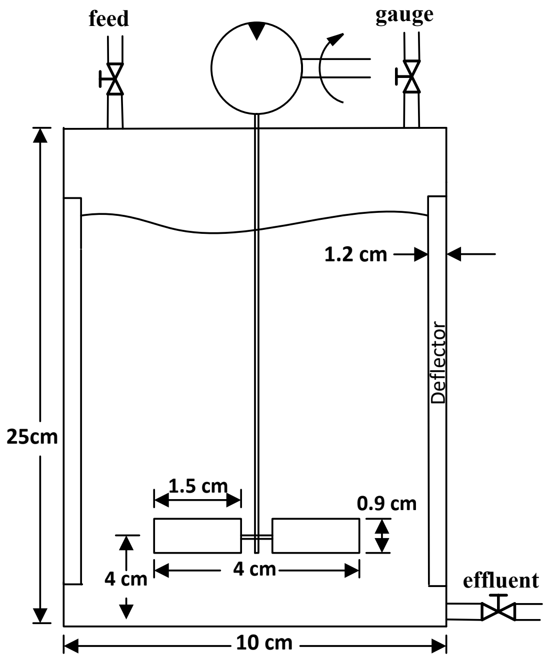

Figure 2. Dimensioning of hydrolysis batch reactor.

3 Results and discussion

3.1 *Geometrical configuration and construction of batch reactor*

Geometrical dimensions of hydrolysis batch reactor were defined employing Mc Cabe mathematical relationships as was described in a previous section. The use of these relationships allows estimating not only the dimensions (height and width) of reactor body and the stirring rod but also the deflector thickness. During batch reactor dimensioning the following considerations were assumed; cylindrical geometry of reactor body, 4 deflectors through transversal periphery as a larger number would create poor mixing zones, 30% volume for headspace considering the formation of steam due to the temperature change, total volume of 2 L, an effluent valve at the bottom for draining and the presence of inlet valve for raw material loading and bearing accessories in the cover of reactor.

From figure 2, it can appreciate that the height and width of reactor body corresponds to 25 and 10 cm, respectively, with a 20 cm of liquid height. The thickness of each deflector was 1.2 cm and the width of agitator blade was defined as 0.9 cm. For stirring rod, design consideration comprises 2 blades per propel.

Reactor body was made with 10 mm thickness stainless steel. At the bottom, ½ in valve was adapted as effluent conduct. The cover of reactor was made with a stainless-steel blind flange, which was interconnected to reactor body by using screws

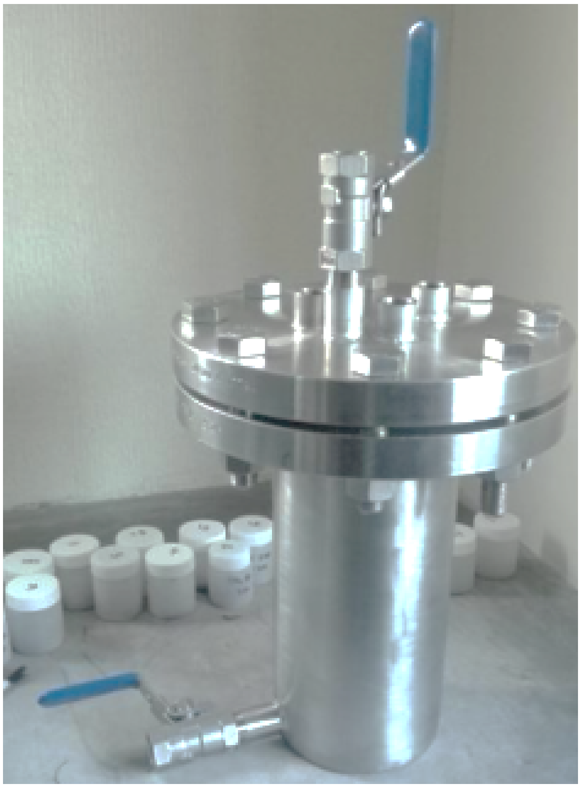

Figure 3. Stainless steel-based hydrolysis batch reactor.

and nuts. To avoid steam leaks at high temperatures, neoprene packing was placed between reactor body and its cover. Figure 3 shows the ensembled hydrolysis batch reactor.

3.2 *Conditioning of batch reactor and CFD analysis of stirring system*

After batch reactor construction, the following stage before its operation for sugars extraction consisted on implementing stirring and heating systems, constructing a metal base to support reactor body and validating the proper distribution of flow inside reactor, so, each component of hydrolysis reactor was drawn using 3D CAD tools (Figure 4).

Drawings were then exported to LS Dyna software to simulate fluid flow profile inside reactor body. The parameters employed for dynamic simulation was: density, 1.004 g / mL; viscosity, 5 centipoise; air heat capacity, 1000 J / kg °C; water heat capacity, 4186 J / kg °C; stirring velocity, 250 rpm; pressure of 1.3 atm. According to the velocity profile shown in Figure 5, the distribution of velocity (mm / s) was not neither homogeneous nor stable, and this can be due to the geometry of thermal resistance which inhibits mass transfer over its surface area. Despite this, there is a semi-homogeneous zone far from resistance where solution was well mixed (Intense blue). The zone appreciated in green colour suggests that mixing process does not well performed, considerably diminishing the linear velocity of solution, however, as can be seen in Figure 5 (left picture), most of the fluid distribution is homogeneous. The use of "U" thermal resistance generates 2 disadvantages: a) reduces mass transfer inside reactor promoting concentration gradient and reducing the overall reaction yield, and b) promotes overheating on resistance surface which can promote an impregnation and burning of solid particles, and the subsequent external resistance damage. To avoid these problems, a new thermal resistance was designed. The new design comprised of a vertical thermal rod with a height of 17 cm and a thickness of 1 cm, coupled to a 1.8 cm thread (Figure 6 (left)), which was adapted to an electronic device that controls reaction temperature and connected into batch reactor. The rod thermal resistance was constructed on stainless steel material and installed from the bottom of the batch reactor.

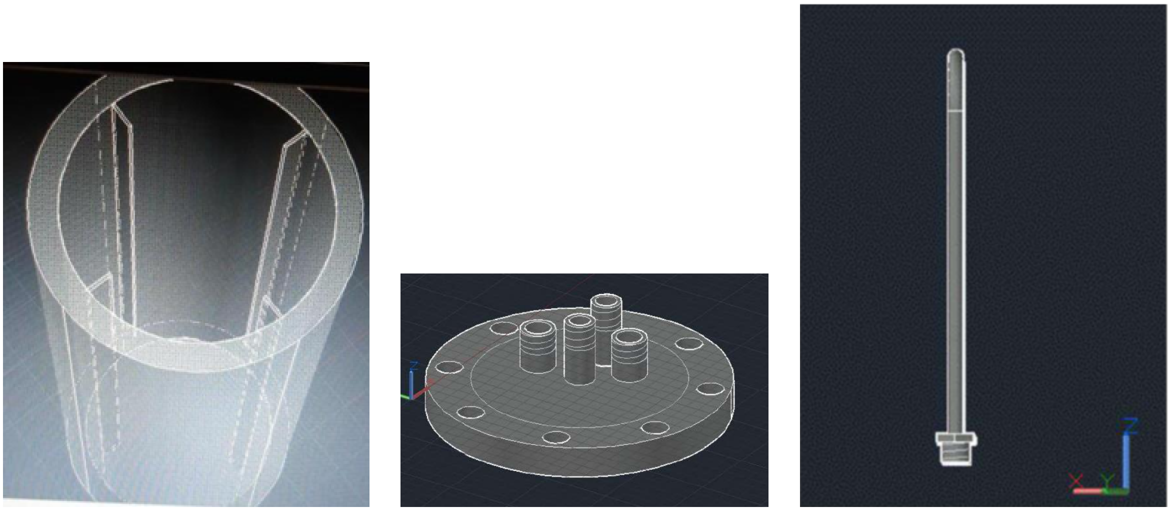

Figure 4. 3D CAD drawings of hydrolysis batch reactor components (From left to right; reactor body, reactor cover and thermometer rod).

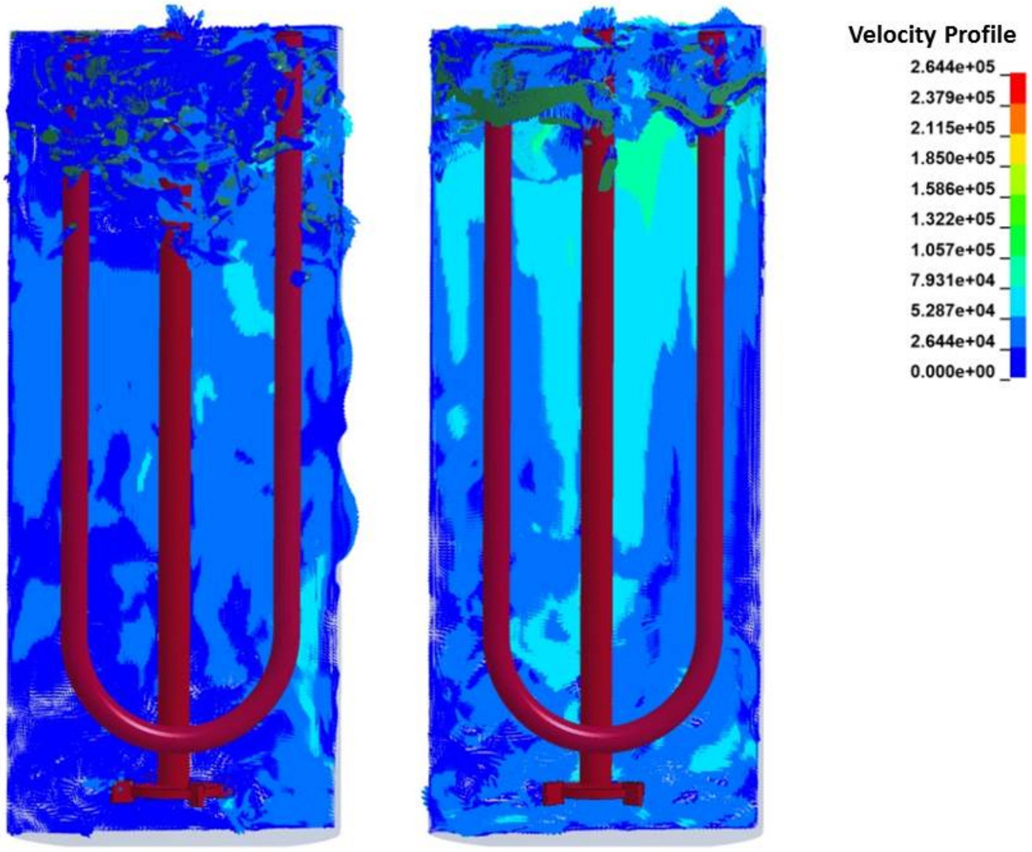

Figure 5. Velocity profile for hydrolysis batch reactor.

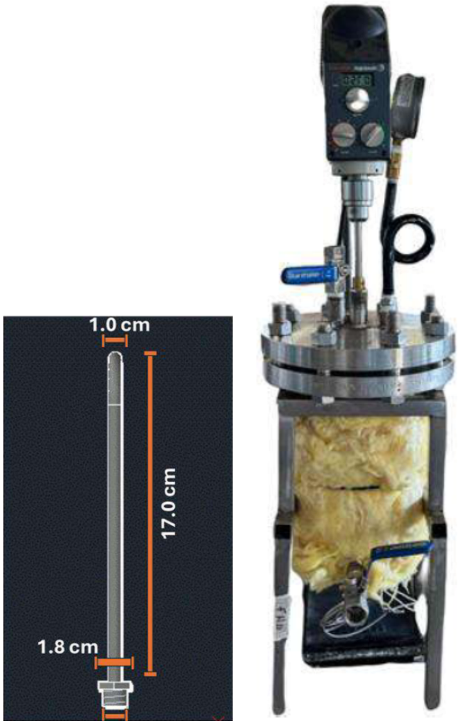

Figure 6. 3D CAD drawing of thermal resistance and the assembled hydrolysis batch reactor.

The assembled hydrolysis batch reactor is presented in Figure 6 (right), as can be seen thermal heating rod was placed at the bottom of the reactor and connected to an electronic device, while stirring system composed by mechanical rotor is placed on reactor cover. The pigtail pressure gauge was adapted near to mechanical rotor, it is expected that hydrolysis reaction operates between 1 and 1.3 atm as internal pressure; this factor will be conditioned on the desired reaction temperature (80 – 130 °C). Since batch reactor was made on stainless steel material, a glass wool cover is necessary to maintain internal temperature by avoiding thermal losses.

The batch reactor has been used for lignocellulose hydrolysis assays employing either direct acid hydrolysis (Rodrigues *et al*., 2010) or enzymatic saccharification (de Godoy *et al*., 2019), producing high sugar yield. The design and construction of heterogeneous batch reactor for hydrolysis purposes date back long years ago, and from this conventional configuration several modifications like plug-flow, percolation, twin gear and shrinking-bed reactors have been proposed in the last decades (Zhou *et al*., 2021). It is well known that batch mode reactor has several advantages; high xylose recovery, easy to operate and maintain as well as simple design and low cost, however, its main disadvantage is the discharge of hydrolysate at the end of the chemical reaction that promotes sugar thermal cracking diminishing saccharides yield. Plug-flow and percolation reactors avoid this trouble and are able to increase sugar recovery; however, they require high energy demand and large water volumes, which make them nowadays un-practicable for industrial purposes (Torget *et al*., 2000). Most recently, twin gear (Ahmed *et al*., 2020) and twin-screw extrusion reactors (Wang *et al*., 2020) have been used to revalorize lignocellulose, the distinctive characteristic of these technologies are the fibrillation and shortening of the biomass fibers by frictional and shearing forces while chemical reagents are added, so, lignocellulose biomass is simultaneously altered by mechanical and chemical interactions. The complexity of this process involves challenges which must be solving like high capital requirement and energy consumption. The abovementioned makes evident the search of economical and feasible reactor configurations that not only need low capital cost but also achieved high sugar recovery.

The hydrolysis batch reactor reported here aims to diminish the construction cost and the operation complexity, since it uses an electronic device to maintain the desired temperature inside reactor and a conventional stirring system comprised by mechanical rotor and stirring rod. Other researchers have studied acid hydrolysis in more complex reactor systems, as example, Rodrigues *et al*. (2010), employed thermal oil as heating source during sugarcane bagasse hydrolysis with sulfuric acid, reaching a xylose yield of 74 %. Similar yield (77 %) was obtained in a semi-pilot batch reactor hydrolyzing rice straw in sulfuric acid solution catalyst (Roberto *et al*., 2003). The achieved sugar yields coupled to low construction cost and simplicity of the process make batch mode the most suitable option for large-scale and industrial applications. However, in accordance with Wright *et al*. (1987), there are some suggestions that could be considered to increase hydrolysis reaction performance: presoaking process to facilitate phases contact and promote protons diffusion inside lignocellulose material, preliminary stage to increase material porosity and the subsequent mass transport and the use of small particles to avoiding mass transfer limitations. In addition, the use of batch reactors allows determining the kinetic behavior of hydrolysis reaction by using simple kinetic equations like conventional Saeman and biphasic models which assume hydrolysis as pseudo-homogeneous reaction with-no mass transfer limitations. Finally, compared with commercial batch reactors, metal-based options are more expensive than reported here. The construction cost of the steel metal-based reactor provided here is quite similar to commercial glass-based reactors which cannot be used at high pressures or dense solid loadings. In addition, commercial reactors employ steam as a heating source depending on the reactor volume, this implies auxiliary services and high energy demand for boiler employment.

3.3 Pineapple crowns acid hydrolysis

The composition of pineapple crown particles employed for hydrolysis reaction was: moisture, 6.88 ± 0.11 %; extractives, 10.19 ± 0.10 %; holocellulose, 63.66 ± 1.61; lignin, 19.28 ± 0.13 %, which is similar to that determined before by Flores-Álamo *et al*. (2024), 69.36 % and 17.85 % for holocellulose and lignin, respectively. In contrast, the holocellulose content of Brazilian pineapple crowns determined by Fernandes Pereira *et al*. (2021) was quite lesser (36.5 %) with similar lignin percentage (24.3 %). High holocellulose content (84.2 %) coupled to low lignin mass fraction (7.4 %) was reported by Johny et al. (2023) working with Indian pineapple wastes. The variety on pineapple crowns composition can be attributed to difference on environmental properties like moisture, soil features and solar radiation, among others. From the high holocellulose content reported here, it is expected that Mexican pineapple crowns generate high sugars yield as shown in Figure 7. The maximum sugar production was 45.50 g / 100 g raw material, observed at 140 minutes, after this time, sugar concentration diminished because of thermal cracking of sugars into aldehydes reaching 35.88 g / 100 g at the end of the experiment. The sugars decomposition after 140 minutes (Figure 7) has also been observed in other works studying dilute sulphuric acid hydrolysis of corn cob (Gutiérrez-López *et al*., 2022) and oat bagasse (Gómora-Hernández *et al*., 2022) at moderate temperatures. In both papers, furfural concentration increased as temperature rises. In literature it has reported that sugars decomposition can be due to the combined impact of acid loading plus operational temperature, however, this last one is the most prominent variable promoting sugars transition into inhibitory aldehydes. In fact, high temperatures (150 – 240 °C) favoured aldehydes production (Yong *et al*., 2022), i.e. monomeric sugars are slowly degraded under 140 °C and short reaction times. Moreover, high severity processes raise sugars dehydration and thus, aldehydes concentration increases (Luo *et al*., 2019). From the abovementioned, is suggested that hydrolysis batch reactor must operate at moderate conditions; low temperature (< 140 °C), short reaction times (180 minutes) and low acid loading (<5 %v) to improve sugars extraction.

Sugar production data was well fitted to Saeman model obtaining the following regression parameters: $k_1 = 0.0168$ min^{-1}, $k_2 = 0.0028$ min^{-1} and $R^2=0.997$. The high value of k_1 rate constant is associated to

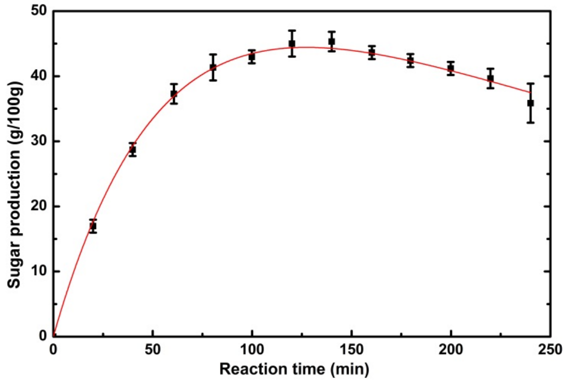

Figure 7. Sugar production data fitted to Saeman kinetic model.

the rapid sugar concentration increase in the first times (t < 140 min). The low value of k_2 is related to the low decomposition of sugars detected after 140 minutes. Previous papers have reported the same kinetic constants trend and related to high sugar yield, i.e. if $k_1 > k_2$, sugar production reaction is favoured (Abril-González *et al.*, 2023; Rafiqul and Sakinah, 2012).

The main idea of construct a hydrolysis batch reactor is to increase the amount of lignocelluloses treated to produce more monomeric saccharides. Recently, our research team has made efforts on improving sugars extraction from different sources, as example, pineapple crowns hydrolysis with sulphuric acid was reported at similar operational conditions to that employed here. In this research, the maximum sugar production yielded at lab-scale was 40.27 g / 100 g at 105 °C (Gutiérrez-López *et al.*, 2022); this value was lower to that obtained here. The difference can be due to the low temperature difference as well as to the improving on solution stirring in the batch reactor. Results are promising for further sugar extraction improving; however, major details on the hydrodynamic behaviour inside reactor are needed to reduce mass transfer limitations.

Conclusions

The reactor design using CFD showed good agitation using 4 baffles with a width of 12 mm, in addition to a 2-blade stirrer with a length of 1.5 cm. A homogeneous velocity profile was not obtained due to the installed thermal resistance that acted as an obstacle. It was assumed that thermal resistance causes good mass transfer to not be achieved, so a new heating method that does not limit stirring will be required. It was possible to operate with a pressure of 1.3 atm and a temperature of 120 °C, in addition to a stirring speed of 250 rpm.

In the hydrolysis kinetics, the maximum production time of reducing sugars was 140 minutes, obtaining 45.5g/100g. The test was carried out for a time of 250 min, at which time the sugar concentration was reduced to 35.88g/g. The production and degradation constants showed that production was maintained over their degradation.

Acknowledgements

Authors thank Tecnológico Nacional de México/Instituto Tecnológico de Toluca for finnancial support (project 19308.24-P).

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