

Article

Design and Performance Evaluation of Batch Reactor for Biomass Valorization: LHW Valorization of Coffee and Peapod Waste

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Abstract: This study presents the design, fabrication, and performance evaluation of a batch reactor for the hydrothermal valorization of coffee and peapod waste biomass. The reactor, designed using Inventor 2023 and analyzed using ANSYS 2023, is capable of operating at elevated temperatures to facilitate the breakdown of lignocellulosic structures and promote the extraction of valuable platform chemicals. Based on the design, the reactor was manufactured, and a set of experiments was performed to test it at different temperatures ranging from 120 to 180 °C, at different times (1–4 h) and with different types of biomass (peapods and coffee cherry waste). The results demonstrate the effectiveness of the reactor in optimizing the conversion of agricultural waste into key compounds such as hydroxymethylfurfural (HMF) and furfural. For peapod biomass, optimal conditions were identified at temperatures between 150 °C and 180 °C, with a reaction time of approximately 1 h yielding up to 72.17%wt total platform chemicals. Coffee cherry biomass showed a different yield profile, with total platform chemical yields reaching 23.56%wt at 180 °C after 4 h of treatment, highlighting the importance of feedstock-specific optimization. The reactor's performance indicates its potential for broader applications in the conversion of various lignocellulosic feedstocks into high-value chemicals, contributing to a more sustainable and circular economy. The presented design and analysis demonstrate the reactor's compliance with necessary characteristics such as temperature, deformation, and stress, ensuring its suitability for the experiments. This study showcases the importance of innovative reactor design in advancing sustainable chemical production from biomass waste, making it a valuable contribution to the field of waste-to-value conversion technologies.



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1. Introduction

Liquid hot water (LHW) pretreatment is a hydrothermal process that transforms lignocellulosic biomass into platform chemicals by utilizing high-pressure water at elevated temperatures, typically between 160 °C and 230 °C [1]. During this process, water remains in its liquid state, allowing it to act both as a solvent and a catalyst. The high temperature and pressure facilitate the hydrolysis of hemicellulose, which is solubilized and removed from the biomass, thereby reducing the recalcitrance of the lignocellulosic structure [2]. This transformation enhances the accessibility of cellulose for subsequent enzymatic hydrolysis, leading to the production of fermentable sugars [3]. The process minimizes the formation of inhibitory degradation products, which can hinder fermentation, making LHW a promising method for biofuel production and the generation of various platform chemicals such as furfural, levulinic acid, and glycerol from biomass feedstocks [4].

The effectiveness of LHW pretreatment is significantly influenced by the operational conditions, including temperature, pressure, and duration of treatment [5]. Maintaining water in its liquid form under high pressure is critical, as it allows for the efficient hydrolysis of biomass components without the need for additional chemicals [6]. This not only simplifies the process but also reduces environmental impacts. The physicochemical changes that occur during LHW pretreatment include the dissolution of hemicellulose, partial removal of lignin, and minimal degradation of cellulose, which collectively contribute to the enhanced hydrolysis of biomass [7]. By optimizing these conditions, LHW can produce a higher yield of platform chemicals, supporting the development of sustainable biorefineries and advancing the bioeconomy.

The design of the reactor is crucial in maintaining the pressurized system and keeping the water in its liquid form during liquid hot water (LHW) pretreatment. A well-designed reactor ensures efficient heat transfer and pressure management, which are essential in achieving the desired temperature and phase of water. Pressurized reactors are particularly important as they allow water to remain in the liquid state, even at high temperatures [8]. This is significant because liquid water acts as an organic solvent that effectively hydrolyzes the lignocellulosic matrix, facilitating the release of fermentable sugars. The ability to operate under pressure not only enhances the efficiency of the pretreatment but also helps in achieving a higher yield of biofuels and other bioproducts [9].

Batch reactors are a fundamental component of liquid hot water (LHW) pretreatment processes, particularly for the conversion of lignocellulosic biomass into biofuels and platform chemicals. In a batch reactor, the biomass and liquid water are combined in a closed system, where they are subjected to high temperatures and pressures. The biomass is heated to the desired temperature, typically between 120 °C and 180 °C, and held for a specific residence time [10]. During this period, the high temperature facilitates the hydrolysis of hemicellulose, which is solubilized and removed from the biomass, thereby enhancing the accessibility of cellulose for subsequent enzymatic hydrolysis. The batch reactor's simplicity allows for precise control over the reaction conditions, including temperature, pressure, and time, which can be adjusted to optimize the pretreatment process for different biomass types.

However, batch reactors also have their limitations. One of the primary drawbacks is their limited throughput, as each batch must be processed sequentially, which can lead to longer processing times and reduced overall efficiency when scaling up for industrial applications. Additionally, the need for cooling and cleaning between batches can add to operational costs and downtime. While batch reactors are excellent for research and development due to their flexibility and ease of operation, they may not be the most economical choice for large-scale production [11]. Therefore, while they play a crucial role in the initial stages of LHW research, there is a growing interest in transitioning to more continuous systems that can handle larger volumes of biomass more efficiently.

In contrast, semicontinuous reactors offer a more streamlined approach to LHW pretreatment. These reactors allow for a continuous feed of biomass while simultaneously removing treated material, which can significantly enhance throughput and reduce processing times compared to batch systems. By maintaining a constant flow of biomass through the reactor, semicontinuous systems can achieve more consistent reaction conditions and better control over the pretreatment process [12]. However, they come with increased complexity in design and operation, requiring careful management of flow rates and reaction parameters to ensure optimal performance. While semicontinuous reactors can improve productivity and efficiency, they also demand more sophisticated control systems and monitoring, which can increase capital and operational costs. As such, the choice between

batch and semicontinuous reactors often depends on the specific production goals and scale of the biomass conversion process.

The design of a well-constructed batch reactor is vital for optimizing the yield of hydrolysis of biomass during liquid hot water (LHW) pretreatment. A properly engineered batch reactor allows for precise control over critical parameters such as temperature, pressure, and residence time, which are essential in maximizing the hydrolysis of hemicellulose and enhancing the accessibility of cellulose. By maintaining the water in its liquid state under high pressure, the reactor facilitates efficient mass transfer and heat distribution, ensuring that the biomass is uniformly treated. This optimization not only improves the overall efficiency of the pretreatment process but also minimizes the formation of inhibitory byproducts, ultimately leading to higher yields of fermentable sugars and platform chemicals. Therefore, investing in the design and engineering of batch reactors is crucial in advancing the economic viability of biomass conversion processes in biorefineries. This article provides a design of a 100 mL batch reactor that allows for the valorization of peapod waste and coffee cherry waste, its FEA and CAD design, as well as a set of experiments that prove the feasibility of the design for different biomasses in different ranges of temperature and time.

2. Materials and Methods

2.1. Reactor Design

In the chemical transformation processes of food products, chemical reactions are carried out in specialized reactors. The design of the reactors usually has specific conditions according to the project, and in some cases, they are even patented for their authenticity. It is very important that the reactor design complies with good performance, which in turn will help with obtaining excellent results. It should be noted that the designs are made on a small scale, scaling up to an industrial scale, considering mass transfer, residence time, and heat transfer, among other parameters. Likewise, the volume was defined by the mixing requirements and the heat transfer by the residence time to achieve the chemical reaction. It is necessary to test the optimum conditions of the design [13].

Eight steps are generally followed when deciding on a design. The first step is the collection of data based on the bibliographic search of the experiments performed and an interactive process between the designer and the researchers. This is followed by the selection of experimental conditions with which to optimize the reactor conversion to provide safety and control for the reactor. The third step corresponds to the determination of the material of construction to perform a preliminary analysis to evaluate the temperature and pressure to which it will be subjected. In the fourth step, the critical parameters of the reactor dimensions are determined. For this, the parameters' intrinsic kinetics, mass transfer rate, heat transfer rate, feed addition rate, and mixing rate must be considered. In the fifth step, a preliminary sizing and cost calculation is performed, where the reactor volume can be determined, and based on this, the design speed can be determined. Additionally, the agitation, how it affects the reactor pressure, and how the selected material acts must be evaluated. We then proceed to estimate the performance and confirm that the design will achieve the ideal conditions. However, it is difficult to test designs at full scale due to the large costs involved. In the seventh step, it is important to optimize the design of the equipment, where it is possible to repeat the previous steps to reduce the cost and time of the process. Finally, we proceed to make the detailed designs with the help of specialized programs and produce the plans on a specific scale where all the characteristics are shown [13].

It is important to note that reactors that operate continuously generate lower costs; however, reactors that operate discontinuously are more useful in smaller-scale experiments

because their operation requires the help of other equipment to fulfill their function. In a batch reactor that is corrosion-resistant, it can be a stainless-steel vessel with heating and cooling coils. In addition, it stands out for heterogeneous reactions, and by implementing stirring, the liquid can be dissipated. In this case, the volume of the reactor can be calculated, and the volume of the liquid that can be used for processing can be specified. Now, it is necessary to calculate the time necessary for the conversion with the mass balance when the volume is the maximum, knowing that it is not constant throughout the process, and in the case of batch reactors, the flows are zero. Another parameter to consider is the temperature, which can be constant or variable, and this will depend on the dissipation rate, in which both speed and concentration will intervene. For the kinetics of batch reactors, two methods can be used: either the differential or the integral method. It can be identified that the differential method directly provides the kinetic parameters, whereas in the integral method, an iterative process is required. Additionally, the differential method requires experimental data for its derivative solution and is a somewhat inexact method. However, a good strategy is a combination of both methods, where the differential method helps us estimate the order of the reaction and the integral method helps us estimate the parameters [14].

There are different types of reactors, as mentioned above; however, they can also be subclassified according to the way the feedstock is processed. It can be single-phase or multiphase processing. In a single phase, it can be a solid, liquid, or gas, and in a multiphase, it can be a combination of them. In batch reactors, their operation is variable due to the concentration and the material contained in the reactor, which allows the kinetic equations to be applied in a simple way. Likewise, the operation is performed in a closed system, and several products can be produced. Another advantage is the low manufacturing cost. Batch reactors have the advantage of processing the material in small quantities for short or long periods of time. Similarly, temperature control is performed with heating jackets, condensers, or pumps [15]. Additionally, the operation is performed in a closed system, and several products can be obtained. Another advantage is the low cost of manufacture, and little by little, the designs have been improved to meet the needs of clean technologies. In batch reactors, the mixing and heat transfer parameters are evaluated in order to achieve a homogeneous dispersion of the components for a good mass transformation in the different phases. There are several types of batch reactors, as shown in Figure 1 [16].

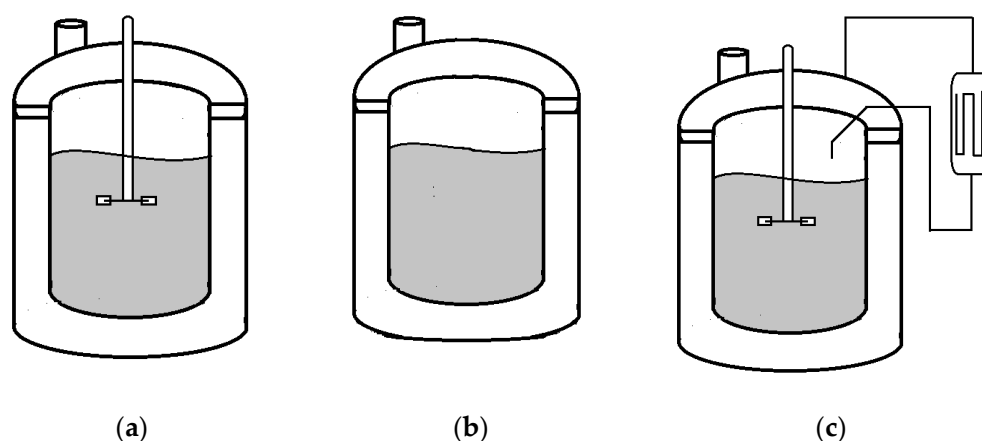


Figure 1. Types of chemical reactors. (a) Jacket with agitation. (b) Jacket without agitation. (c) Jacket with agitation and condenser.

The important variables involved in the process are the following:

- Residence Time Distribution: Detailed explanation of the time that the elements spend inside the reactor [15].

- Heat Transfer: The reaction rate and how temperature affects it must be considered. Reactors are generally evaluated for their performance, phase presentation, and temperature control for heat transfer [15].
- Mass Balance: This allows for an overview of the materials entering and leaving the system for the design of the chemical reactor under the concept of mass conservation. Mass balances can be based on the total mass or focus on a chemical element or compound, and depending on this, the chemical equation is established. Therefore, the integral mass balance starts by identifying the limits of the system and everything that can influence it and will vary depending on the type of chemical reactor [15].

When selecting the reactor type, a preliminary selection should be made based on reaction kinetics and process parameters. Before making a selection, it is very important to define factors for selection that reduce the cost and increase the benefit. The factors are technical, economic, and social. The technical factors depend on two chemical and physical factors that are in charge of controlling the yield and quality. The economic factors include capital and operating costs, and finally, the social factors are those that affect society and do not have a monetary value. In addition, before the reactor is selected, the reaction conditions, the heat of reaction, the need for agitation and reaction mixture, and finally, the reactor construction material must be known [17]. Chemical reactors are responsible for ensuring a mixture of the reacting materials, providing sufficient time to achieve the desired reaction, and finally, allowing the conditions of pressure and temperature to give rise to the degree of the proposed reaction [18].

The equations that apply in the process start with Equation (1) describing the mass balance. However, in batch reactors, there is no inflow or outflow, so the input and output matter rates are zero. By replacing the terms, Equation (2) can be obtained [11].

$$\left(\begin{array}{c} \text{Accumulated} \\ \text{Matter Rate} \end{array} \right) = \left(\begin{array}{c} \text{Input Matter} \\ \text{Rate} \end{array} \right) - \left(\begin{array}{c} \text{Output Matter} \\ \text{Rate} \end{array} \right) \pm \left(\begin{array}{c} \text{Reacting Matter} \\ \text{Rate} \end{array} \right) \quad (1)$$

$$V \frac{dC}{dt} = -kC^n V \quad (2)$$

where V is reactor volume, k is organic matter removal rate, C is organic matter concentration, and n is the order of kinetics of the equation. When the order of the equation is 1, Equation (3) can be obtained.

$$C = C_0 e^{-kt} \quad (3)$$

The following is the energy balance approach followed by the equations describing the area and volume of the reactor [19], where $L = 1.5 D$.

$$\frac{dT}{dt} = \frac{UA}{\rho_r C_{p_r} V} (T_j - T) + \frac{\Delta H_{r_{SH}} V}{\rho_r C_{p_r} V} + \frac{\mu N^2 V}{\rho_r C_{p_r} V} \quad (4)$$

$$A = \pi D L \quad (5)$$

$$V = \frac{\pi}{4} D^2 L \quad (6)$$

2.2. Experimental Design

To analyze the efficiency of the reactor in the range of LHW, a set of experiments was made at different temperatures and times evaluating the resistance of the nylon jacket and the stainless-steel reactor. The experiments are presented below (Table 1) and in all the cases, the biomass used was dried and meshed into a 2 mm particle size, and ratios of 1:10 (biomass/water) were used, filling the reactor up 1/3, hence adding 3 g of dried biomass and 27 mL of deionized water.

Table 1. Experimental design for reactor optimization.

Experiment	Biomass	Time (h)	Temperature (°C)
1	Peapod	1	120
2	Peapod	1	130
3	Peapod	1	140
4	Peapod	1	150
5	Peapod	1	160
6	Peapod	1	170
7	Peapod	1	180
8	Peapod	2	180
9	Peapod	3	180
10	Peapod	4	180
11	Coffee Cherry	1	120
12	Coffee Cherry	1	130
13	Coffee Cherry	1	140
14	Coffee Cherry	1	150
15	Coffee Cherry	1	160
16	Coffee Cherry	1	170
17	Coffee Cherry	1	180
18	Coffee Cherry	2	180
19	Coffee Cherry	3	180
20	Coffee Cherry	4	180
21	Coffee Cherry	5	180

After the reactions, the solid and liquid were filtered and weighed, in order to identify the mass balance and the gas production and possible leaks. The liquid phase was identified by HPLC-IR with a Hitachi Elite LaChrom (Tokyo, Japan), a Hitachi L-2490 refraction index detector at 40 °C, a SHODEX Sugar SH1821 column at 60 °C, 0.005 M H₂SO₄ for the mobile phase, and a flow rate of 0.5 mL/min. Levulinic acid, furfural, xylose, glucose, mannose, arabinose, HMF, and formic acid were quantified as the hydrothermal platform chemicals produced via batch reactors.

The yields of platform chemicals (PC) were calculated as shown in Equation (7):

$$\% \text{ Yield}_{PC} = \frac{(\text{grams of platform chemicals obtained})}{(\text{grams of lignocellulosic structure in biomass})} \times 100\% \quad (7)$$

The grams of lignocellulosic structure in biomass (Table 2) corresponds to the results obtained by applying the procedure described in “Methods for Dietary Fiber, Neutral Detergent Fiber, and Non-starch Polysaccharides in Relation to Animal Nutrition” in the *Journal of Dairy Science* [20].

Table 2. Characterization of initial biomass.

Essay	Coffee Cherry	Peapods
Lignin (%wt)	13.7	5.0
Cellulose (%wt)	27.6	20.2
Hemicellulose (%wt)	12.5	17.4
Transformable lignocellulosic structure in biomass (%wt)	40.1	37.6

2.3. Reactor Design

Based on the chemical reactor design approach, an overview of the ongoing project on the valorization of agricultural wastes in hydrothermal processes such as LHW is presented. Additionally, the experimental conditions that the reactor needs to meet with respect to temperature can be evidenced. A temperature range of 120 to 180 °C is handled. This process was selected because preliminary experimental tests were carried out to determine

the behavior of the biomasses (coffee and peapod) in the first hydrothermal process and to define the outlook for the following hydrothermal processes, which are the purpose of the project. Due to the fact that these were preliminary tests, it was required that the quantities of biomass and products were used in smaller quantities; therefore, it was necessary that the reactor in which we worked had a small-scale volume.

To carry out the proposed experiments, the service provider designed and built a chemical reactor with stainless steel. This reactor had a capacity of 100 mL, a maximum temperature of 180 °C, and consisted of several parts. Figure 2 shows the breakdown of the mechanical parts of the reactor. Figure 2a shows the main parts of the reactor, and Figure 2b shows the breakdown of the parts of the reactor lid, which is the one that exerts the force so that the reactor does not leak.

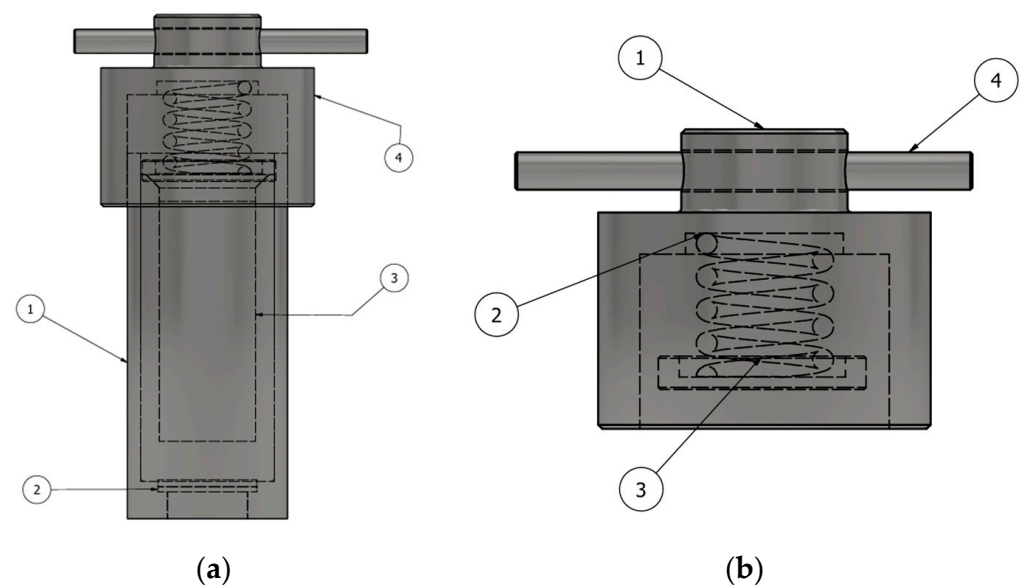


Figure 2. Parts of chemical reactor. (a) Main parts: 1. metal container; 2. metal bottom plug; 3. Teflon container; 4. metal cover. (b) Metal cover: 1. metal structure; 2. spring; 3. metal spring base; 4. torque tool.

Figure 3 shows the chemical reactor used and its dimensions. The reactor works as follows:

1. The components, in this case the biomass with water, are placed in the Teflon jacket and sealed with the Teflon lid.
2. The Teflon container is inserted into the steel container.
3. Fit the steel container with the steel lid, making sure that the metal base of the spring is centered with the Teflon lid.
4. Tighten the steel cap with the steel container, making sure that the thread is completely secured. Because the spring exerts great pressure to adjust the reactor, it is necessary to use a press and an adjusting tool.
5. Once the reactor has been adjusted, it should be placed in a preheated laboratory oven in which the temperature and time are programmed according to the experiment.
6. At the end of the time, the reactor is allowed to cool for a few minutes outside the oven and then placed in a bath with cold water to lower its temperature. Once it has cooled to room temperature, the Teflon container should be opened and removed to complete the chemical experiment.

Additionally, a finite element analysis is performed for the polytetrafluoroethylene (PTFE) vessel where the behavior is observed with respect to a structural analysis. Polytetrafluoroethylene is a thermoplastic that does not react with other chemical reactions apart from in exceptional cases. Its properties [21] can be seen in the Figure 4 below.

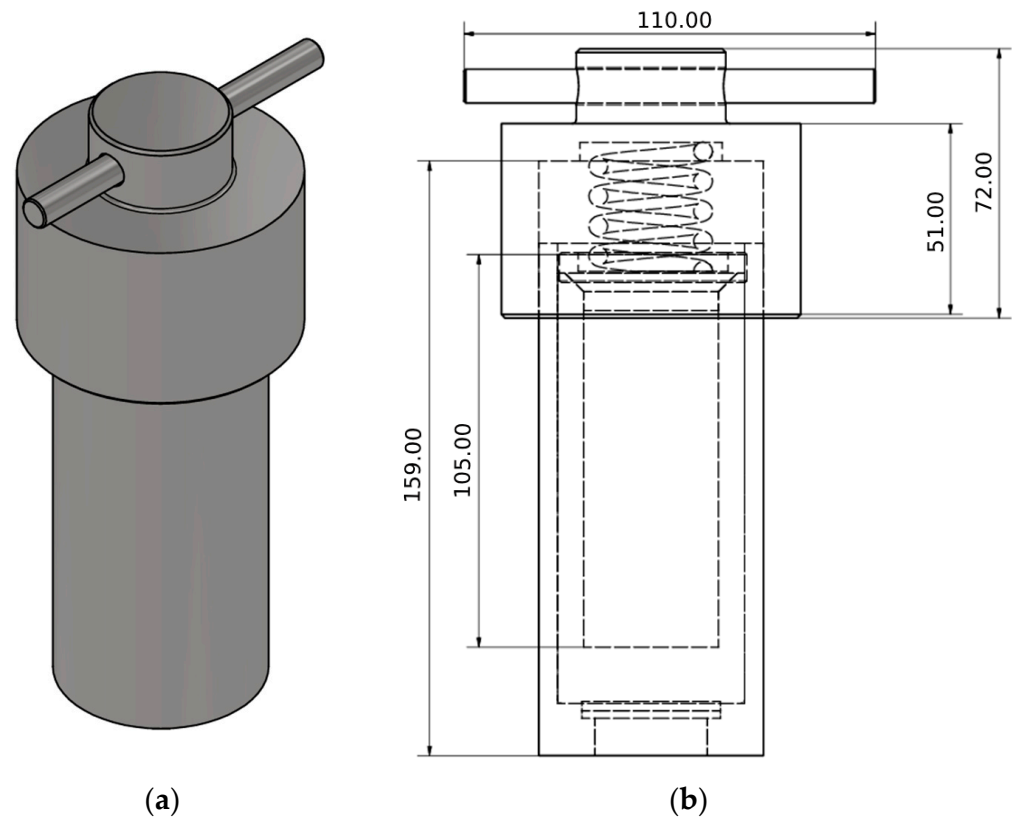


Figure 3. Chemical reactor. (a) Side view of the reactor. (b) Dimensions of the reactor.

Figure 5 shows the results obtained in the structural analysis. In the image, a shows the behavior of the total deformation of the part. It is shown that the largest deformation is 2.6597×10^{-9} m and the smallest is 0 m. However, the maximum values represented in red color are presented in minimum percentage in the upper and inner part of the vessel. It can also be observed that the deformation decreases from the top to the bottom, as shown by the colimetry varying from 2.3641×10^{-9} m to the minimum. In addition, the deformations presented by the material under the conditions of force and support are minimal. Image b shows the results of the Von Misses stress, where the maximum stress is 197.69 Pa, and it is present in the lower part of the cylinder in a minimum percentage. Additionally, it is evident that in the internal part of the vessel, the stress is decreasing from 176.17 Pa to the minimum stress of 4.0553 Pa. In the external part of the vessel, it is evidenced that 80% presents stresses ranging between 133.14 Pa and 25.57 Pa. The maximum stress of PTFE is 0.750 GPa; therefore, the material resists the stresses that are being exposed in the process because it does not exceed the maximum stress of the process. The material reaches temperatures of 260 °C in its extreme condition; however, in the test it is being taken to 180 °C and would be performing its function.

Properties of Outline Row 3: PTFE			
	A	B	C
1	Property	Value	Unit
2	Material Field Variables	Table	
3	Density	2.2	g cm ⁻³
4	Isotropic Secant Coefficient of Thermal Expansion		
5	Coefficient of Thermal Expansion	12	C ⁻¹
6	Isotropic Elasticity		
7	Derive from	Young's Mod...	
8	Young's Modulus	550	MPa
9	Poisson's Ratio	0.46	
10	Bulk Modulus	2.2917E+09	Pa
11	Shear Modulus	1.8836E+08	Pa
12	Tensile Yield Strength	196	MPa
13	Compressive Yield Strength	24.1	MPa
14	Isotropic Thermal Conductivity	0.25	W m ⁻¹ K ⁻¹

Figure 4. Polytetrafluoroethylene properties.

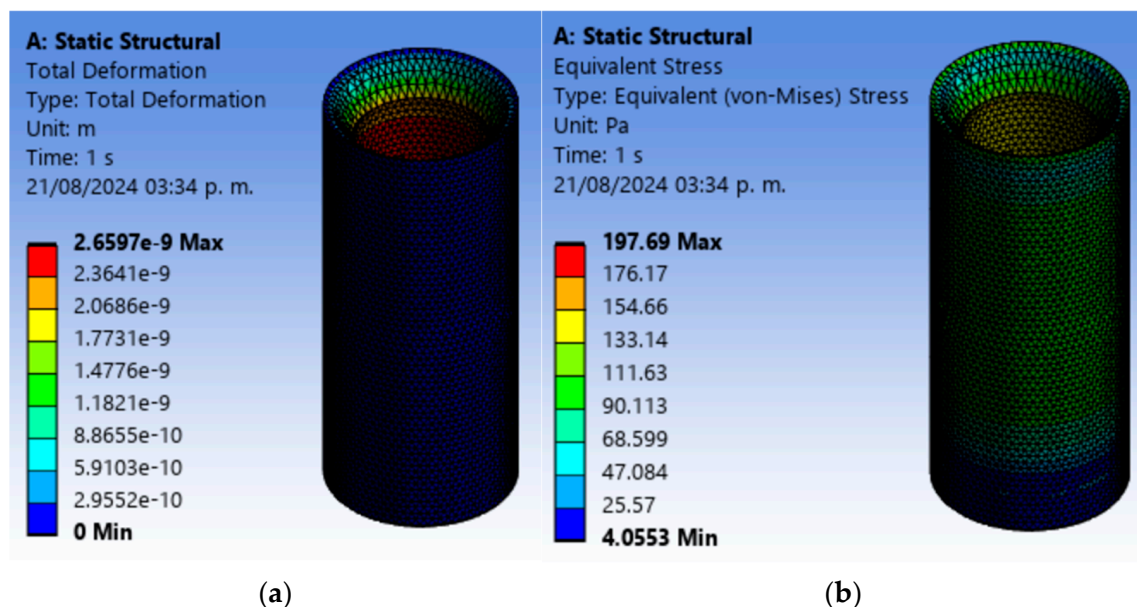


Figure 5. Results of structural analysis. (a) Results of total deformation. (b) Results of equivalent elastic strain.

3. Results and Discussion

Hydrothermal Valorization with Designed Batch Reactor

A preliminary test following the mass balance was performed with the coffee sample in order to identify possible leaks in the designed reactor, and the results are presented in Table 3.

It can be seen that in all the cases under 170 °C, there was a similar amount of solid fraction. Considering that it started with 3 g of coffee cherry waste, it shows consistency that the change was not due to reactor malfunctions but rather due to the fact that at higher temperatures, processes of valorization and transformation are going on inside the reactor. As for the liquid and gas fraction, a similar behavior took place: there was not a big change under 180 °C; the amount of liquid fraction and gas fraction remained the same, showing that there were no leaks and that the reactor was withstanding the pressure of the water. At higher temperatures over 180 °C, chemical transformations took place, which transformed

the biomass, and extracted unstable and volatile compounds into the liquid fractions which later evaporated into the gas fraction. This also shows that even though there were no leaks, the reactor was functional and allowed valorization to take place.

Table 3. Evaluation of solid, liquid, and gas fraction after hydrothermal reaction using the designed batch reactor.

Temperature (°C)	Time (h)	Solid Fraction (g)	Liquid Fraction (g)	Gas Fraction (g)
120	1	2.789	22.920	4.296
130	1	2.689	22.918	4.393
140	1	2.650	22.830	4.520
150	1	2.633	22.835	4.532
160	1	2.590	22.873	4.537
170	1	2.546	22.904	4.550
180	1	2.498	22.890	4.502
180	2	2.148	20.310	7.852
180	3	1.995	16.871	12.005
180	4	1.895	14.281	14.105
180	5	1.390	15.120	13.489

In order to identify said transformation and valorization, quantification of the platform chemicals was performed, and the results are presented in Table 4.

Table 4. Results of biomass valorization implementing the designed reactor.

Biomass	Time (h)	T (°C)	Total CP (%)	Sugars (%)	FA (%)	L.A (%)	HMF (%)	Furfural (%)
Peapod	1	120	36.903	36.903				
Peapod	1	130	37.228	37.228				
Peapod	1	140	37.610	37.610				
Peapod	1	150	56.265	56.265				
Peapod	1	160	53.023	53.023				
Peapod	1	170	62.774	62.089		0.865		
Peapod	1	180	72.170	70.994		1.176		
Peapod	2	180	53.361	46.704	4.242	1.885	0.531	
Peapod	3	180	17.936	6.276	7.212	1.986	1.342	1.120
Peapod	4	180	23.655	2.217	15.187	2.206	1.829	2.215
Coffee								
Cherry	1	120	0.167	0.167				
Coffee								
Cherry	1	130	0.213	0.213				
Coffee								
Cherry	1	140	0.323	0.323				
Coffee								
Cherry	1	150	2.347	2.347				
Coffee								
Cherry	1	160	2.391	2.391				
Coffee								
Cherry	1	170	2.654	2.654				
Coffee								
Cherry	1	180	2.709	2.709				
Coffee								
Cherry	2	180	3.209	3.209				
Coffee								
Cherry	3	180	7.502	4.855	1.785	0.862		
Coffee								
Cherry	4	180	23.56	6.036	12.38	3.775	0.656	0.718
Coffee								
Cherry	5	180	21.59	8.554	10.02	4.919		

F.A: formic acid. L.A: levulinic acid.

The results obtained from the 100 mL batch reactor demonstrate the effective conversion of lignocellulosic biomass into platform chemicals (PC), particularly from peapod and coffee cherry biomass. The data indicate varying yields of total platform chemicals, sugars, and other components such as furfural and hydroxymethylfurfural (HMF) across different temperatures and treatment times. The reactor's performance is evidenced by the significant increase in total PC percentages, especially at higher temperatures (e.g., 150 °C and above), which correlates with enhanced hydrolysis and transformation processes. This suggests that the reactor is well-designed for optimizing the extraction of valuable chemicals from biomass.

The mechanisms underlying these results involve hydrolysis, where cellulose and hemicellulose in the biomass are broken down into fermentable sugars. This process is typically facilitated by thermal treatment, which disrupts the lignocellulosic structure, making it more accessible to enzymatic action [22]. As the temperature increases, the rate of hydrolysis also rises, leading to higher yields of sugars and platform chemicals. For instance, at 150 °C, the total PC yield from peapod reaches 56.265%wt, indicating effective conversion. The subsequent temperatures show variations in yields, likely due to the formation of by-products and the degradation of sugars into other compounds like HMF and furfural, which are also valuable platform chemicals. The transformation of sugars into platform chemicals involves further chemical reactions, including dehydration and rearrangement [23].

For example, HMF is typically formed from the dehydration of fructose or glucose, while furfural is derived from pentoses present in hemicellulose [24]. The presence of these compounds in the results indicates that the reactor not only facilitates hydrolysis but also promotes subsequent reactions that enhance the overall yield of platform chemicals. The data show that at higher temperatures and extended reaction times, the formation of these chemicals increases, reflecting the reactor's capability to optimize conditions for both hydrolysis and subsequent transformations. Overall, the results highlight the reactor's efficiency in converting lignocellulosic biomass into a range of valuable chemicals, aligning with the principles of biorefinery processes. The ability to produce platform chemicals like HMF and furfural from peapod and coffee cherry biomass demonstrates the potential for sustainable chemical production. This process not only contributes to reducing reliance on fossil fuels but also supports the development of a bio-based economy, as outlined in recent literature on biomass valorization and platform chemical production.

The results obtained from the conversion of peapod biomass in a hydrothermal reactor reveal significant potential for producing valuable platform chemicals (PC). The data indicate that total PC yields increase markedly with rising temperature and reaction time, peaking at 150 °C with a yield of 56.265%wt. This demonstrates the reactor's effectiveness in facilitating the breakdown of lignocellulosic structures, which is critical in maximizing the extraction of fermentable sugars and other valuable compounds from peapod biomass. As the temperature increased from 120 °C to 150 °C, the hydrolysis process became more efficient, leading to a substantial release of sugars. At 150 °C, the total PC yield was significantly higher than at lower temperatures, highlighting the importance of thermal treatment in enhancing the hydrolysis of cellulose and hemicellulose present in the peapods. This thermal disruption of the biomass structure allows for greater accessibility to enzymatic action, which is essential in converting complex carbohydrates into simpler sugars that can be further transformed into platform chemicals. The subsequent transformation of these sugars into platform chemicals, such as HMF and furfural, is also noteworthy.

At higher temperatures, particularly at 170 °C and 180 °C, the formation of HMF and furfural increased, indicating that the reactor not only promotes hydrolysis but also facilitates the necessary chemical reactions for producing these valuable compounds. The

presence of these chemicals in the results underscores the reactor's dual functionality in both hydrolysis and subsequent transformation, which are vital in achieving high overall yields of platform chemicals. However, extending the reaction time at 180 °C led to a decrease in total PC yields, suggesting that prolonged exposure to high temperatures may result in the degradation of sugars and platform chemicals [25]. The optimal conditions for peapod biomass conversion appear to be about 150–180 °C for approximately 1 h, balancing the formation of desired products while minimizing undesirable side reactions. These findings highlight the reactor's efficiency in converting peapod biomass into a range of valuable chemicals, positioning it as a promising option for sustainable biomass valorization. Further research could focus on refining these parameters to enhance the recovery of platform chemicals from this agricultural waste stream.

The results for coffee cherry biomass indicate a promising potential for converting this agricultural waste into valuable platform chemicals using a hydrothermal reactor. At 180 °C, the total PC yield reached 23.56%wt, demonstrating the reactor's effectiveness in optimizing the extraction of chemicals from coffee cherry biomass. This significant yield highlights the reactor's capability to facilitate the conversion of lignocellulosic material into useful products, which is crucial in developing sustainable biorefinery processes. The hydrolysis of cellulose and hemicellulose in coffee cherry biomass is enhanced by thermal treatment, which disrupts the structural integrity of the lignocellulosic matrix. As the temperature increased from 120 °C to 180 °C, the total PC yield rose from a mere 0.167% to 23.56%wt. This increase indicates that higher temperatures promote more efficient hydrolysis, leading to the release of fermentable sugars that can be further transformed into platform chemicals. The ability to extract these sugars effectively is essential in maximizing the overall yield of valuable products.

Following hydrolysis, as previously mentioned, the released sugars undergo further transformation reactions, such as dehydration and rearrangement, to produce platform chemicals like hydroxymethylfurfural and furfural. At 180 °C, the yields for these compounds were 0.656%wt for HMF and 0.718%wt for furfural, indicating that the reactor not only facilitates hydrolysis but also promotes subsequent reactions that enhance the overall yield of platform chemicals. This dual functionality underscores the reactor's efficiency in converting coffee cherry biomass into a range of valuable chemicals. Extending the reaction time at 180 °C appeared to be beneficial in maximizing total PC yields, as evidenced by the increase from 2.709%wt at 1 h to 23.56%wt at 4 h, and 21.59% at 5 h.

These findings suggest that prolonged exposure to high temperatures allows for more complete conversion of the biomass, although careful optimization is necessary to avoid potential degradation of the products. Overall, the optimal conditions for coffee cherry biomass conversion seem to be about 180 °C for 4–5 h, highlighting the potential for utilizing this agricultural waste stream in sustainable chemical production. Further research could focus on refining these parameters to enhance the recovery of valuable chemicals from coffee cherry biomass and the design of a semicontinuous reactor to increase yields.

4. Conclusions

In conclusion, the results from the hydrothermal conversion of peapod and coffee cherry biomass demonstrate the feasibility and effectiveness of using the designed 100 mL batch reactor for producing valuable platform chemicals. The reactor operates efficiently at elevated temperatures, facilitating the hydrolysis of lignocellulosic structures and promoting the subsequent transformation of released sugars into key compounds such as hydroxymethylfurfural (HMF) and furfural. The significant yields achieved, particularly with peapod biomass, highlight the reactor's capability to optimize the extraction of valuable chemicals from agricultural waste by keeping water liquid at a high range of

temperatures. This technology not only supports the sustainable valorization of biomass but also offers a practical solution for integrating waste materials into biorefinery processes, contributing to a more sustainable and circular economy. Overall, the reactor's performance indicates its potential for broader applications in the conversion of various lignocellulosic feedstocks into high-value chemicals, making it a promising tool for advancing sustainable chemical production.

The presented hydrothermal reactor effectively facilitates the breakdown of lignocellulosic structures due to its ability to maintain high pressures for water inside of the reaction to keep its liquid state, leading to efficient hydrolysis, enhanced yields of fermentable sugars, and subsequent transformation into platform chemicals. For peapod biomass, optimal conditions were identified at temperatures between 150 °C and 180 °C, with a reaction time of approximately 1 h yielding up to 72.170%wt total platform chemicals. This indicates that thermal treatment plays a crucial role in maximizing the extraction of valuable compounds from peapod biomass. In contrast, coffee cherry biomass showed a different yield profile, with total PC yields reaching 23.56%wt at 180 °C after 4 h of treatment. This suggests that longer reaction times at elevated temperatures may be beneficial for maximizing the conversion of coffee cherry biomass, highlighting the importance of optimizing reaction parameters for different feedstocks.

Additionally, the design of a 100 mL reactor is presented. Its computer aided design was obtained using the inventor program version 2023 and its finite element analysis using Ansys version 2023. It can therefore be concluded that the vessel material complied with the necessary characteristics, such as temperature, deformation, and stress, for the experiments presented. It showed no leaks in the range of LHW, and proper functioning in subcritical water conditions that allow for hydrothermal processes to take place.

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References

1. Singh, A.; Tsai, M.-L.; Chen, C.-W.; Singhanian, R.R.; Patel, A.K.; Tambat, V.; Dong, C.-D. Role of hydrothermal pretreatment towards sustainable biorefinery. *Bioresour. Technol.* **2023**, *367*, 128271. [[CrossRef](#)] [[PubMed](#)]
2. Deng, W.; Feng, Y.; Fu, J.; Guo, H.; Guo, Y.; Han, B.; Jiang, Z.; Kong, L.; Li, C.; Liu, H.; et al. Catalytic conversion of lignocellulosic biomass into chemicals and fuels. *Green Energy Environ.* **2023**, *8*, 10–114. [[CrossRef](#)]
3. Tanis, M.H.; Wallberg, O.; Galbe, M.; Al-Rudainy, B. Lignin Extraction by Using Two-Step Fractionation: A Review. *Molecules* **2023**, *29*, 98. [[CrossRef](#)] [[PubMed](#)]

4. Chakraborty, P.; Kumar, R.; Chakraborty, S.; Saha, S.; Chattaraj, S.; Roy, S.; Banerjee, A.; Tripathy, S.K.; Ghosh, A.K.; Jeon, B.-H. Technological advancements in the pretreatment of lignocellulosic biomass for effective valorization: A review of challenges and prospects. *J. Ind. Eng. Chem.* **2024**, *137*, 29–60. [\[CrossRef\]](#)
5. Antczak, A.; Szadkowski, J.; Szadkowska, D.; Zawadzki, J. Assessment of the effectiveness of liquid hot water and steam explosion pretreatments of fast-growing poplar (*Populus trichocarpa*) wood. *Wood Sci. Technol.* **2022**, *56*, 87–109. [\[CrossRef\]](#)
6. Scapini, T.; dos Santos, M.S.; Bonatto, C.; Wancura, J.H.; Mulinari, J.; Camargo, A.F.; Klanovicz, N.; Zabet, G.L.; Tres, M.V.; Fongaro, G.; et al. Hydrothermal pretreatment of lignocellulosic biomass for hemicellulose recovery. *Bioresour. Technol.* **2021**, *342*, 126033. [\[CrossRef\]](#) [\[PubMed\]](#)
7. Li, M.; Cao, S.; Meng, X.; Studer, M.; Wyman, C.E.; Ragauskas, A.J.; Pu, Y. The effect of liquid hot water pretreatment on the chemical–structural alteration and the reduced recalcitrance in poplar. *Biotechnol. Biofuels* **2017**, *10*, 237. [\[CrossRef\]](#) [\[PubMed\]](#)
8. Yan, L.; Ma, R.; Li, L.; Fu, J. Hot Water Pretreatment of Lignocellulosic Biomass: An Effective and Environmentally Friendly Approach to Enhance Biofuel Production. *Chem. Eng. Technol.* **2016**, *39*, 1759–1770. [\[CrossRef\]](#)
9. Patel, B.; Guo, M.; Izadpanah, A.; Shah, N.; Hellgardt, K. A review on hydrothermal pre-treatment technologies and environmental profiles of algal biomass processing. *Bioresour. Technol.* **2016**, *199*, 288–299. [\[CrossRef\]](#) [\[PubMed\]](#)
10. Imman, S.; Laosiripojana, N.; Champreda, V. Effects of Liquid Hot Water Pretreatment on Enzymatic Hydrolysis and Physico-chemical Changes of Corncobs. *Appl. Biochem. Biotechnol.* **2018**, *184*, 432–443. [\[CrossRef\]](#)
11. Foutch, G.L.; Johannes, A.H. Reactors in Process Engineering. In *Encyclopedia of Physical Science and Technology*; Elsevier: Amsterdam, The Netherlands, 2003; pp. 23–43. [\[CrossRef\]](#)
12. Barroso, T.L.C.T.; da Rosa, R.G.; Sganzerla, W.G.; Castro, L.E.N.; Maciel-Silva, F.W.; Rostagno, M.A.; Forster-Carneiro, T. Hydrothermal pretreatment based on semi-continuous flow-through sequential reactors for the recovery of bioproducts from jaboticaba (*Myrciaria cauliflora*) peel. *J. Supercrit. Fluids* **2022**, *191*, 105766. [\[CrossRef\]](#)
13. Sinnott, R.; Towler, G. Design of Reactors and Mixers. In *Chemical Engineering Design*; Elsevier: Amsterdam, The Netherlands, 2020; pp. 1039–1146. [\[CrossRef\]](#)
14. Ravi, R. Reactor Design—General Principles. In *Coulson and Richardson’s Chemical Engineering*; Elsevier: Amsterdam, The Netherlands, 2017; pp. 1–101. [\[CrossRef\]](#)
15. Nanda, S. *Reactors and Fundamentals of Reactors Design for Chemical Reaction*; Department of Pharmaceutical Sciences: Rohtak, India, 2008.
16. Winterbottom, J.M.; King, M.B. *Reactor Design for Chemical Engineers*, 1st ed.; Routledge (Publishers) Ltd.: Birmingham, UK, 1999; Volume 1.
17. Safety in Chemical Reaction Engineering. In *Modeling of Chemical Kinetics and Reactor Design*; Elsevier: Amsterdam, The Netherlands, 2001; pp. 910–1033. [\[CrossRef\]](#)
18. Towler, G.; Sinnott, R. *CHEMICAL ENGINEERING DESIGN: Principles, Practice and Economics of Plant and Process Design*, 1st ed.; Elsevier: San Diego, CA, USA, 2008; Volume 1.
19. Ahmed, G.S.; Humadi, J.I.; Aabid, A.A. Mathematical Model, Simulation and Scale up of Batch Reactor Used in Oxidative Desulfurization of Kerosene. *Iraqi J. Chem. Pet. Eng.* **2021**, *22*, 11–17. [\[CrossRef\]](#)
20. Van Soest, P.J.; Robertson, J.B.; Lewis, B.A. Methods for Dietary Fiber, Neutral Detergent Fiber, and Nonstarch Polysaccharides in Relation to Animal Nutrition. *J. Dairy Sci.* **1991**, *74*, 3583–3597. [\[CrossRef\]](#)
21. Matweb. Overview of Materials for Polytetrafluoroethylene (PTFE), Extruded. Available online: <https://www.matweb.com/search/DataSheet.aspx?MatGUID=4e0b2e88eeba4aaeb18e8820f1444cdb&ckck=1> (accessed on 12 August 2024).
22. Yuan, Y.; Jiang, B.; Chen, H.; Wu, W.; Wu, S.; Jin, Y.; Xiao, H. Recent advances in understanding the effects of lignin structural characteristics on enzymatic hydrolysis. *Biotechnol. Biofuels* **2021**, *14*, 205. [\[CrossRef\]](#) [\[PubMed\]](#)
23. Arias, K.S.; Velly, A.; Climent, M.J.; Iborra, S. Valorization of biomass platform molecules through one-pot cascade processes using heterogeneous catalysts. *Tetrahedron Green Chem* **2024**, *3*, 100037. [\[CrossRef\]](#)
24. Zhao, Y.; Lu, K.; Xu, H.; Zhu, L.; Wang, S. A critical review of recent advances in the production of furfural and 5-hydroxymethylfurfural from lignocellulosic biomass through homogeneous catalytic hydrothermal conversion. *Renew. Sustain. Energy Rev.* **2021**, *139*, 110706. [\[CrossRef\]](#)
25. Ruiz, H.A.; Conrad, M.; Sun, S.N.; Sanchez, A.; Rocha, G.J.M.; Román, A.; Castro, E.; Torres, A.; Rodríguez-Jasso, R.M.; Andrade, L.P.; et al. Engineering aspects of hydrothermal pretreatment: From batch to continuous operation, scale-up and pilot reactor under biorefinery concept. *Bioresour. Technol.* **2020**, *299*, 122685. [\[CrossRef\]](#) [\[PubMed\]](#)

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