

Original Research

Valorization of Agro-Industrial Residues into Furfural via Catalytic Pyrolysis

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Abstract

Pyrolysis is a promising technology for converting biomass waste into bio-oil, a liquid product that can serve as feedstock for fuels and high-value chemicals. In this study, bio-oil was produced via catalytic pyrolysis of pear pulp waste, a byproduct from juice and jam manufacturing, using a fixed-bed reactor. Initial non-catalytic experiments were conducted under nitrogen and vacuum atmospheres at temperatures ranging from 300 to 450°C for 15 minutes to evaluate the effect of temperature on bio-oil yield. The resulting bio-oils were



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analyzed by gas chromatography-mass spectrometry (GC-MS) to identify and quantify the chemical compounds. Based on the identified optimal temperature, catalytic pyrolysis experiments were conducted using mesoporous xCe/SBA-15 catalysts with $x = 0, 0.1$, and 0.2 Ce/Si molar ratios. The catalysts were characterized by XRD, BET surface area analysis, SEM, Raman spectroscopy, and TGA. Among them, the 0.1Ce/SBA-15 catalyst demonstrated the highest activity for furfural production, achieving 15% selectivity and a relative area of 48%. Furfural is a valuable platform chemical used in the synthesis of bioplastics and as a precursor for herbicides, fungicides, and insecticides.

Keywords

Biomass; catalytic pyrolysis; furfural; mesoporous materials; pear pulp waste; platform compounds

1. Introduction

The development of a culture of consumption based on the use of fossil resources is responsible for the enormous amount of greenhouse gases released into the atmosphere and for the increasing generation of poorly biodegradable waste [1]. The accumulation of agro-industrial residues at different stages of production processes is currently a global problem, since in most cases, these residues are not properly processed or disposed of, contributing to environmental pollution [2]. These residues have a high potential for various processes, including the manufacturing of new products and the addition of value while recovering altered ecological conditions [3, 4]. Organic waste, particularly that of plant origin, is a valuable source of lignocellulosic biomass. This biomass, primarily composed of cellulose, hemicellulose, and lignin, can be converted into value-added products, though efficient technologies are required to overcome the challenges associated with its conversion [5, 6]. In this sense, thermochemical and biocatalytic processes allow the valorization of residues into a variety of oxygenated compounds [7-10].

Among biomass-derived chemicals, furfural stands out as one of the most valuable due to its high reactivity, which makes it an excellent platform molecule for the synthesis of a wide range of industrially important compounds [11]. Currently, furfural is used in numerous applications, including as a selective extraction agent, solvent, vulcanizing agent, and fungicide. It also serves as a flavoring agent in various food products and beverages, both alcoholic and non-alcoholic, and is a key ingredient in commercial herbicides, insecticides, antiseptics, and disinfectants [11-15]. Additionally, furfural is used in the production of pharmaceuticals, cosmetics, fragrances, and household cleaners and detergents [11, 12].

Furfural is produced through the mineral acid hydrolysis of pentosans in lignocellulosic biomass, followed by subsequent dehydration of the released pentosan sugars [16, 17]. The process involves hemicellulose fractionating into xylose, which is then dehydrated to form furfural. Production typically begins with a physical pretreatment of the biomass, followed by the addition of acid in a reactor. The reaction is maintained at an optimal temperature via steam injection, and furfural is extracted from the aqueous stream by steam distillation. Finally, the residual solids are recovered, dried, and used as fuel.

Many types of catalysts have been reported for furfural production, and they can be broadly classified into homogeneous and heterogeneous catalysts [18]. Homogeneous catalysts, which are uniformly distributed in the solvent, exhibit high catalytic performance and remain in the same phase as the reactants; however, challenges related to separation and recovery significantly limit their use [19]. Heterogeneous catalysts, in contrast, exist in a different phase from the reaction medium, which facilitates their separation, recycling, and reuse, particularly when solid biomass residues are absent after the reaction. Still, they often require external forces to enhance contact with reactants [16, 17].

Pyrolysis (Py) is an up-and-coming technology for converting biomass residues into value-added compounds [20]. This thermochemical process transforms solid biomass into three main products: a liquid phase (bio-oil), a solid carbonaceous residue (char), and a non-condensable gas with minimal greenhouse gas emissions. Bio-oil is a complex mixture of organic compounds formed through the thermal degradation of cellulose, hemicellulose, lignin, and other biomolecules naturally present in plant biomass. The pyrolysis of biomass involves numerous simultaneous and consecutive reactions, making it challenging to elucidate the detailed reaction mechanisms required for intrinsic kinetic modeling [21]. As a result, various modeling approaches have been proposed, including simplified first-order models that condense the complexity of multiple reactions [22]. Due to the chemical heterogeneity of biomass, composed of diverse structural components with different morphologies, and the influence of process variables, catalysts are often required to improve bio-oil quality, reduce oxygen content, and selectively produce compounds with higher economic value [21]. Catalyst design plays a crucial role in biomass pyrolysis. Properties such as the type and distribution of acidic (Brønsted and Lewis) and basic sites, porosity, metal incorporation, and textural characteristics directly influence catalytic performance [23]. Key structural parameters, including pore size and distribution, specific surface area, crystalline phases, and support architecture, must be carefully tailored, as they significantly affect catalytic activity, selectivity, and target-product yield [23]. Moreover, the nature of the biomass feedstock imposes specific requirements on catalyst functionality. Depending on its composition, structure, and functional groups, different catalytic properties may be required, necessitating customized designs to maximize the production of selected compounds [24]. For practical and industrial applications, catalysts must also exhibit high resistance to deactivation, low regeneration cost, and strong selectivity toward desired products [25]. The broad spectrum of catalytic materials available offers extensive opportunities to control the complex reaction pathways of biomass pyrolysis. A wide variety of catalysts, including zeolites, alkaline and alkaline earth metal oxides, transition-metal oxides, carbon-based materials, and mesoporous structures, have been explored for the pyrolysis of diverse biomass feedstocks [26, 27]. Among these, zeolite-based catalysts have received considerable attention due to their well-defined porous structures and tunable acidity, parameters that strongly influence product selectivity and coke formation [28].

A study [28] involving different types of mesoporous MCM-41 with various metal contents (Fe, Al, or Cu) demonstrated that the presence of mesoporous Al-MCM-41 can significantly improve the quality of the bio-oil produced during in-situ pyrolysis of biomass by increasing the yield of desirable fractions.

Chen et al. [29] experimentally investigated the fast pyrolysis of different biomasses (corn cob, wheat straw, and cotton stalk) using solid catalysts composed of titanium (TiN, TiO₂, and TiOSO₄) and metal nitrides (MoN, GaN, and VN) for furfural production by Py-GC/MS. The results indicated

that TiN and GaN promoted furfural formation mainly through the direct decomposition of oligosaccharides. When comparing the three biomass residues, a higher furfural yield was obtained from corn cob due to its higher holocellulose content, while greater furfural selectivity was achieved from wheat straw. Fast pyrolysis of biomass impregnated with zinc chloride (ZnCl_2) has also shown promise for producing furfural and acetic acid [30]. Maximum furfural yields were obtained from corn cob impregnated with at least 15 wt.% ZnCl_2 at approximately 340°C, achieving yields above 8 wt.%, compared to only 0.49 wt.% for raw corn cob. Branca et al. similarly performed pyrolysis of ZnCl_2 impregnated corn cobs and reported a maximum furfural yield of 6% using a ZnCl_2 concentration of 2 to 5% by weight [31].

Metal oxides, such as TiO_2 , ZrO_2 , MgO , CaO , and CeO_2 , represent another important class of catalysts currently explored for bio-oil upgrading [32-34]. These materials exhibit diverse catalytic functionalities: for instance, MgO effectively reduces the oxygen content in bio-oil, while CeO_2 promotes the formation of ketonic compounds.

Among mesoporous materials, SBA-15 has emerged as an auspicious catalyst support due to its high specific surface area, excellent thermal stability, and tunable pore structure, all of which enhance biomass conversion by improving the accessibility and distribution of active sites [33, 35].

Integrating large mesopores with catalytic functionalities into SBA-15-based materials offers a strategic advantage, with potential to develop novel and efficient catalysts or catalyst supports [35]. Their inherent versatility, stemming from the presence of mineral components that may serve as active sites, can be further tuned through appropriate thermal or chemical activation, thereby modifying their surface chemistry, porosity, and metal anchoring capacity. In this context, dispersing active metals onto mesoporous support enhances catalytic activity and mitigates deactivation under severe reaction conditions [35, 36]. Moreover, the rational design of catalysts with strong metal-support interactions and effective confinement of metal nanoparticles is essential to maintain stability and reactivity throughout the pyrolysis process [35].

From a mechanistic perspective, furfural formation during catalytic pyrolysis of lignocellulosic biomass is mainly linked to the decomposition of the hemicellulosic fraction, where xylan-derived intermediates undergo dehydration and rearrangement reactions to form furfural. Acidic catalysts promote these pathways by stabilizing reactive intermediates and enhancing selective dehydration, while suppressing competing fragmentation and polymerization reactions. Therefore, the interaction between biomass-derived pyrolysis vapors and tailored acidic catalytic sites is a key factor in directing the complex reaction network toward selective furfural production [37].

Despite considerable advances, developing efficient catalytic systems for converting biomass into high-value chemicals remains a significant challenge and a research priority. In this work, the fast pyrolysis of pear pulp residues was experimentally investigated using Ce/SBA-15 catalysts with different Ce/Si molar ratios to enhance the selective production of furfural.

2. Experimental

2.1 Raw Material Characterization

Pear pulp (PP) waste from the manufacture of jams was provided by DULCOR S.A. (Province of Cordoba). Before the experiments, the PP waste (Figure 1) was ground and placed in an oven at 150°C for 24 h to remove moisture. The elemental composition (C, H, N, and S) of pear pulp was

determined using a LECO CHNS-932 elemental analyzer, based on the dynamic combustion method in accordance with ASTM standard procedures.



Figure 1 PP waste from fruit processing for the manufacture of jams.

The compositional analysis of pear pulp was conducted following standardized protocols. The extractives content was determined using hot water (TAPPI T207) and ethanol (TAPPI T264) extractions. Lignin content was quantified using the Klason method (TAPPI T222), while cellulose was determined by the Kürschner-Höffner method. Hemicellulose content was estimated by mass difference between the major structural components. Additionally, moisture content and ash content were measured in accordance with TAPPI T210 and TAPPI T211, respectively.

Thermogravimetric analysis (TGA) was carried out using a Mettler Toledo TGA/SDTA851e/SF/1100°C thermobalance. Approximately 10 mg of dried PP sample was subjected to thermal decomposition under a nitrogen atmosphere (flow rate: 50 mL/min), from room temperature up to 600°C at a constant heating rate of 10°C/min. This small sample size was chosen to minimize heat and mass-transfer limitations. Mass loss and temperature data were continuously recorded throughout the experiment to assess the thermal behavior of the biomass.

2.2 Synthesis of the Catalysts

Ce/SBA-15 materials were synthesized by a hydrothermal process [30]. In a typical procedure, 4 g of triblock copolymer Pluronic P-123 (Aldrich) were dissolved in 138 mL of distilled water with vigorous stirring. Then, the necessary amount of cerium (III) nitrate hexahydrate ($\text{Ce}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}$, Aldrich) was added together with 11.4 g of 2 M HCl solution (Cicarelli). Finally, 8.4 g of tetraethoxysilane (TEOS, Aldrich) were added dropwise, and the resulting mixture was stirred at 40°C for a day. After this, the pH was adjusted to 4 with 2 M ammonia hydroxide solution (Cicarelli), and the suspension was stirred for an additional 4 h before the hydrothermal treatment at 100°C for 24 h under static conditions. The obtained solid was filtered under reduced pressure, washed with distilled water until neutral pH, and dried overnight at 60°C. The powder was calcined in an

oven at 550°C for 8 h, with a heating rate of 1°C/min. The catalysts were named (x)Ce/SBA-15, where x is the theoretical Ce/Si molar ratio.

2.3 Physicochemical Characterization

X-ray diffraction (XRD) patterns were recorded at ambient temperature using Panalytical Empyrean equipment, CuK radiation, and a 3D PIXEL detector. Transmission electron microscopy (TEM) images were acquired employing FEI TECNAI G2 (200 kV) equipment. Scanning electron microscopy (SEM) images of the synthesized powders were obtained using an Inspect S50 microscope. The textural properties of the catalyst samples were evaluated by nitrogen adsorption–desorption isotherms using a Quantachrome Autosorb-1 surface area analyzer. Before analysis, the samples were degassed under vacuum at 150°C for 4 h to remove any physisorbed species. The specific surface area was calculated using the Brunauer–Emmett–Teller (BET) method. Fourier-transform infrared (FT-IR) spectroscopy was performed using the KBr pellet technique on a Thermo Scientific Nicolet iS10 Spectrometer with Smart OMNI-Transmission accessory to identify functional groups and structural characteristics. To investigate the nature of surface acid sites, FT-IR spectroscopy of adsorbed pyridine was conducted, allowing differentiation between Brønsted and Lewis acid sites.

2.4 Pyrolysis Experiments

Pyrolysis experiments were conducted in a horizontal fixed-bed quartz reactor housed within a laboratory-scale tubular furnace (Figure 2). Reactions were performed both in the absence and in the presence of the catalyst. The reactor system included an auxiliary quartz reactor with a constriction, allowing physical separation of the biomass and catalyst bed. Once the desired reaction parameters—temperature, pressure, and carrier gas flow were stabilized, the auxiliary reactor was introduced into the heated zone to initiate pyrolysis.

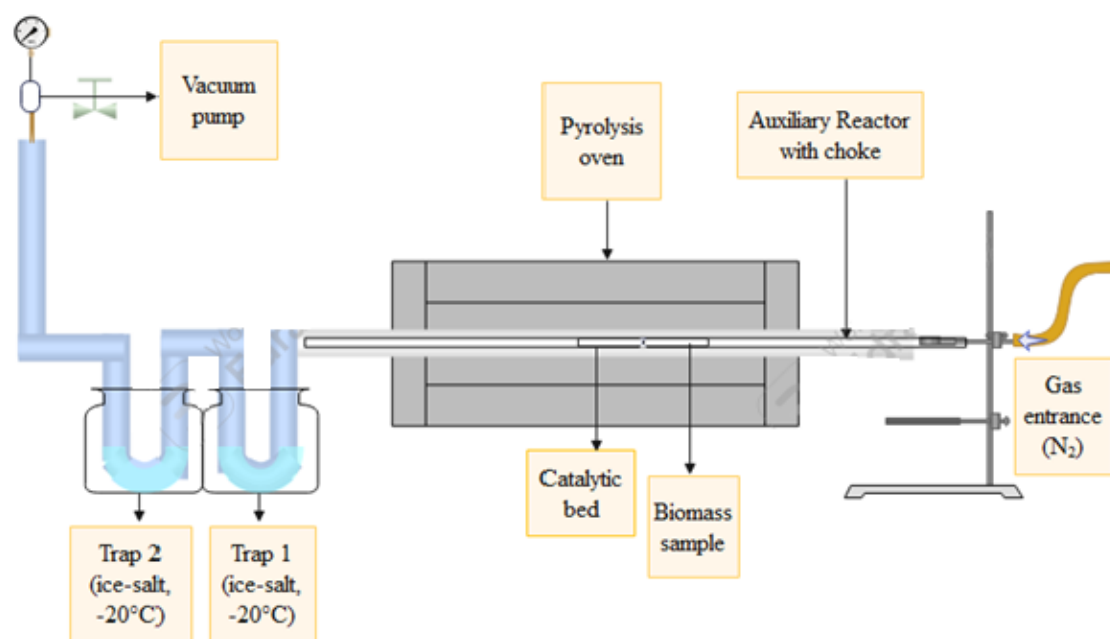


Figure 2 General scheme of the pyrolysis equipment.

One side of the reactor was connected to a vacuum pump, enabling short residence times of the pyrolysis vapors (<1 s). The volatile products were rapidly condensed in two refrigeration traps maintained at -20°C to prevent secondary reactions. Ultra-high purity nitrogen was used as the carrier gas at a constant flow rate of 30 mL·min⁻¹, ensuring the effective transport of volatiles to the condensation unit. For each run, approximately 3 g of dried pear pulp and 10 wt.% of catalyst were loaded into the reactor.

A catalyst loading of 10 wt.% was selected based on previous studies carried out by the authors using similar agro-industrial biomasses, in which this proportion consistently provided an adequate balance between catalytic activity and operational stability. Although different catalyst loadings were not examined in the present work, the 10 wt.% loading was adopted as a reference condition, as it has been shown to promote the dehydration pathways associated with furfural formation. Future investigations will evaluate the influence of varying catalyst amounts to determine whether selectivity and catalytic performance can be further optimized.

Pyrolysis was carried out at 300, 350, 400, and 450°C. This temperature range was selected based on the thermal behavior of hemicellulose-rich biomass, where hemicellulose is reported to decompose predominantly between 200–300°C, while cellulose degrades between ≈250–380°C [38]. Several studies have shown that the formation of furanic compounds, including furfural, is maximized at intermediate temperatures (≈320–380°C). In contrast, higher temperatures favor secondary cracking to light oxygenates and gases, reducing the selectivity toward furfural [39, 40]. Evaluating 300–450°C, therefore, allowed the identification of conditions that balance extensive hemicellulose conversion with high furfural selectivity.

All experiments were performed under reduced pressure (360 mmHg). The reaction time was fixed at 15 min, with time zero defined as the moment the auxiliary reactor was inserted into the pyrolysis zone. Upon completion, both the reactor and the condensation traps were cooled to ambient temperature before disassembly. Each experiment was conducted in triplicate under identical conditions to ensure reproducibility.

Condensed bio-oil was recovered from the refrigeration traps using pro-analysis acetone, and the solvent was subsequently removed under reduced pressure using a rotary evaporator. The chemical composition of the resulting bio-oil was analyzed by gas chromatography coupled with mass spectrometry (GC–MS) using a Shimadzu QP2020 instrument equipped with an HP-5ms capillary column (30 m × 0.25 mm i.d.). Compound identification was based on spectral comparison with the NIST mass spectral library, using a match quality threshold of ≥80%.

Product yields were determined gravimetrically. The liquid and solid fractions were quantified directly by weight, while the gas fraction was estimated by mass balance difference. The biochar and bio-oil yields were evaluated by measuring the mass differences between the sample carrier and the condensation traps. Equations (1), (2), and (3) were used to calculate the products yields.

$$Y_{\text{bio-oil}} (\text{wt}\%) = (M_{\text{bio-oil}}/M_o) \cdot 100 \quad (1)$$

$$Y_{\text{bio-char}} (\text{wt}\%) = (M_{\text{bio-char}}/M_o) \cdot 100 \quad (2)$$

$$Y_{\text{gas}} (\text{wt}\%) = 100 - [Y_{\text{bio-oil}} + Y_{\text{bio-char}}] \quad (3)$$

Where M_o is the initial mass of the biomass sample, $M_{\text{bio-char}}$ is the mass of the solid product after the reaction, and $M_{\text{bio-oil}}$ is the mass of the liquid product.

The selectivity of different bio-oil product fractions (S_i) was calculated using the following Eq (4):

$$S_i = \frac{A_i * Y_{bio-oil}}{100} \quad (4)$$

Where A_i is the relative area of i product in bio-oil from pyrolysis of 3 g PP waste at $T = 350^\circ\text{C}$, $Y_{bio-oil}$ is the yield of the target liquid product, and M is the total.

The relative area of different bio-oil product fractions (A_i) was calculated using the following Eq (5):

$$A_i = \frac{a_i}{a_n} * 100\% \quad (5)$$

Where a_i is the mass of i product in bio-oil from pyrolysis of 3 g PP waste at $T = 350^\circ\text{C}$, and a_n is the total of the liquid product produced in the biomass pyrolysis.

2.5 Characterization of Non-Condensable Gases from Non-Catalytic Pyrolysis

During the pyrolysis process, the gaseous products from the system were sampled using specific gas bags for correct storage. Two gas samples were collected from each of three runs at 350°C .

To determine the composition of the non-condensable gases produced during pyrolysis, a gas chromatograph, model HP 5890 Series II Plus, with a thermal conductivity detector (TCD) was used. A Molsieve column of 30 m and 0.53 mm diameter was used. The operating temperatures were 35, 50 and 200°C for the injector, oven, and detector, respectively. A flow of N_2 as a carrier gas of 10 mL/min was used, and 2 mL of gas were injected.

Gas samples were collected only at 350°C for optimal liquid yield.

3. Results and Discussion

3.1 Biomass Characterization

3.1.1 Elemental Analysis

The elemental composition of the starting biomass is shown in Table 1. As expected, carbon is the main element (wt.%), followed by H (wt.%), while there are minimal differences in N and S contents.

Table 1 Elemental analysis of pear pulp (PP).

	Carbon (wt.%)	Hydrogen (wt.%)	Nitrogen (wt.%)	Sulfur (wt.%)	Oxygen (wt.%)
PP	42.18	5.94	0.41	0.87	50.6

The residues contain a small amount of N and S, which is advantageous because it minimizes corrosion problems associated with the formation of NO_x and acids, and prevents acid rain [41].

3.1.2 Analytical Methods

The differences in their chemical structures led to distinct chemical reactivities, making the relative composition of the biomass a crucial factor in the design of a process.

In the Analytical Procedure, the presence of soluble material (extractives) was determined (with water according to TAPPI 207) [41, 42] and (with ethanol according to TAPPI 264) [43]; the cellulose content was determined by the Kurschner-Hoffer method [44], the lignin content by the Klason method (according to TAPPI 222 standard) [45] and the hemicellulose content were calculated by difference.

The components comprising the extractives/volatiles include both water and ethanol-soluble components. Water-soluble compounds include nonstructural sugars and proteins, and ethanol-soluble components are typically represented by chlorophyll and waxes.

In this case, these components make up most of the biomass composition and could have a significant influence on what ultimately becomes the optimal conversion process.

3.1.3 Thermogravimetric Analysis

The compositions of the residues are also determined by thermogravimetric analysis (TGA-DTG) as an alternative method and to assess the reliability of wet methods. Thermogravimetric analysis, particularly the derivative thermogravimetric (DTG) curve, provides insight into the thermal decomposition behavior of biomass by identifying distinct devolatilization stages [46]. Under inert conditions, the pyrolytic degradation of lignocellulosic biomass typically proceeds through four main stages: initial moisture loss, followed by sequential decomposition of hemicellulose, cellulose, and lignin, the three principal structural components of biomass [35].

The thermogravimetric curves obtained (Figures 3a, 3b) indicate that the devolatilization of the pear pulp residue initiates at approximately 150°C, with the most significant mass loss occurring between 200 and 350°C. Beyond 350°C, the TGA profile shows a marked reduction in the rate of weight loss, which continues more gradually up to 400°C (Figure 3a). An initial weight loss observed around 120°C corresponds to the evaporation of residual moisture. Overall, more than 70 wt.% of the sample was volatilized by 500°C, indicating a high content of thermally labile components.

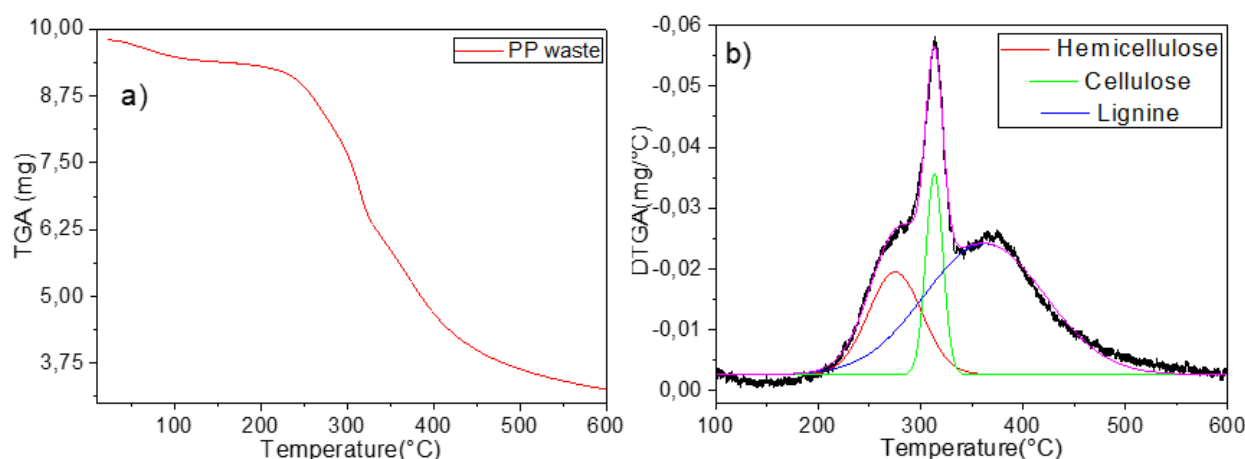


Figure 3 Thermogravimetric Analysis (TGA) of PP waste (a), Differential Thermal Analysis (DTA) curve with the deconvolution calculation (b).

The derivative thermogravimetric analysis curves (Figure 3b) revealed three main thermal degradation events associated with the principal biomass constituents. Deconvolution of the DTG signal allowed differentiation of the overlapping degradation peaks: hemicellulose decomposition occurred in the range of 200–300°C; a sharp peak observed between 250–350°C was attributed to α -cellulose; and a broader shoulder in the 350–500°C range was associated with lignin degradation. The relative intensities and integrated areas under these peaks correlate with the proportions of each macromolecular component (Table 2). Based on DTG deconvolution, hemicellulose was identified as the predominant component, with cellulose and a lower lignin content also contributing to the thermal degradation profile [47].

Table 2 Structural chemical composition of biomass.

Methodology	Cellulose	Hemicellulose	Lignin
Analytical	17.06 $\pm$ 0.01	20.73 $\pm$ 0.01	8.99 $\pm$ 0.01
TGA	22.53 $\pm$ 3.5E-5	27.17 $\pm$ 3.5E-5	50.30 $\pm$ 3.5E-5

The difference between the analytical and TGA values is due to interference from extractives: pure biomass was used for the thermogravimetric analysis, whereas biomass free of extractives was used in the analytical methods.

3.2 Catalyst Physicochemical Characterization

Catalyst design plays a critical role in steering pyrolysis reactions toward targeted products. Key parameters include the nature, strength, and distribution of Brønsted and Lewis acid/base sites, as well as textural properties such as porosity, specific surface area, and pore size distribution. The incorporation of metal species further modifies the catalytic surface, introducing new active sites and influencing the electronic and structural environment. Additionally, the crystalline phase and the catalyst's overall structural integrity are essential design considerations. These physicochemical characteristics collectively govern the catalytic activity and selectivity, ultimately impacting the yield and composition of the desired pyrolysis products [48].

The pore ordering of the synthesized materials (0.1Ce/SBA-15, 0.2Ce/SBA-15, and SBA) was corroborated by low-angle X-ray diffraction. The patterns in Figure 4a show three well-resolved peaks at 2θ of 1.0, 1.5, and 1.8, indexed to planes (1 0 0), (1 1 0), and (2 0 0), characteristic of the hexagonal lattice symmetry of SBA-15 ($p6mm$). Therefore, the incorporation of cerium into the silica framework successfully yielded a highly ordered mesostructured material with a two-dimensional hexagonal pore arrangement for all Ce/Si molar ratios studied. Nonetheless, it is essential to note that the peak intensity decreased with increasing Ce content, indicating that the ordered mesostructure is gradually distorted by the incorporation of heteroatoms into the mesoporous framework [36]. In this sense, both the d_{100} interplanar distance and the cell parameter (a_0) increase as the cerium molar rate concentration increases (see Table 3). However, the values of d_{100} and a_0 are comparable to those reported for SBA-15-type materials in the literature [49].

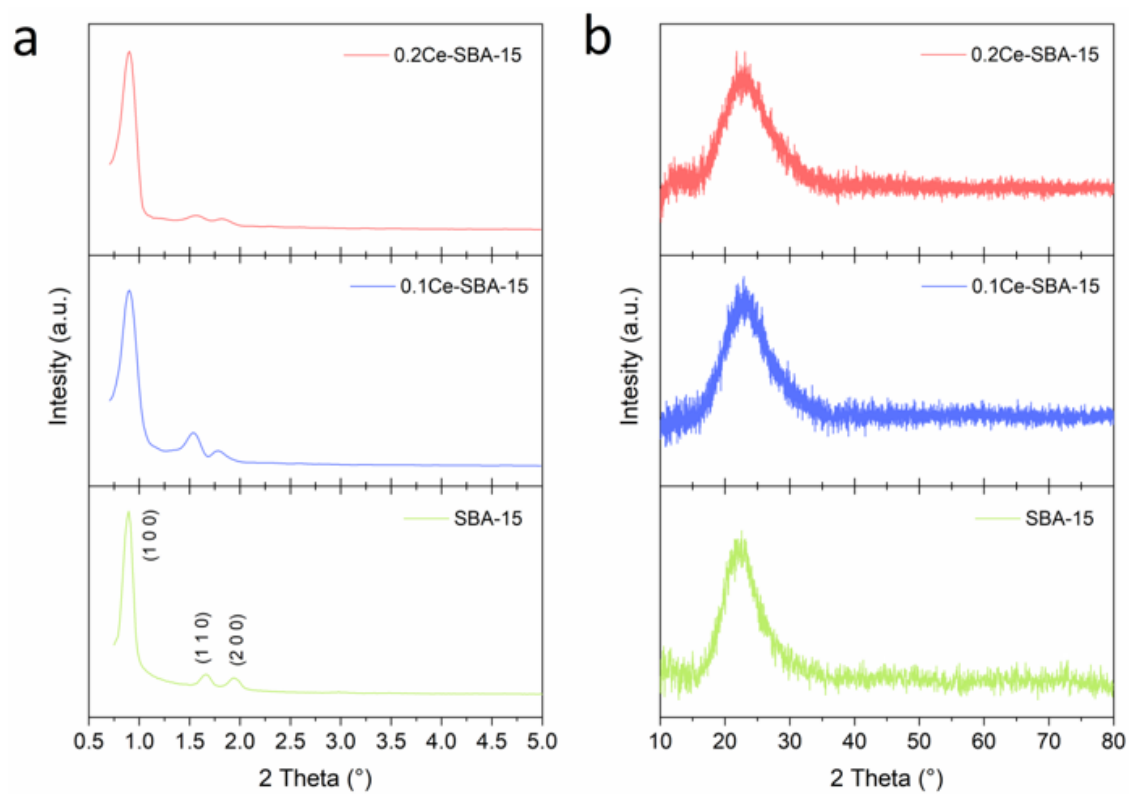


Figure 4 Low-angle (a) and high-angle (b) X-ray diffraction patterns of the synthesized materials (SBA-15, 0.1Ce/SBA-15, and 0.2Ce/SBA-15).

Table 3 Chemical composition, textural and structural properties of the synthesized catalysts.

Catalyst	Ce content ^a (wt.%)	Theoretical Si/Ce molar ratio	d_{100} ^b (Å)	a_0 ^c (nm)	Specific area ^d (m ² /g)	Pore diameter ^e (nm)	Wall thickness ^f (nm)	Pore volume (cm ³ /g)
SBA-15	-	-	95.84	11.07	636	6.03	5.04	0.84
0.1Ce/SBA-15	0.73	10	96.47	11.1	745	8.7	2.4	1.11
0.2Ce/SBA-15	1.42	5	97.86	11.3	781	8.9	2.4	1.17

^a Determined by ICP. ^b Calculated from DRX data ($n\lambda = 2d \sin(\theta)$). ^c Lattice parameter ($a_0 = (2/3^{1/3}) d_{100}$).

^d Calculated by the BET method. ^e Calculated by the BJH method. ^f a_0 – pore diameter.

High-angle X-ray diffraction patterns of all the samples (Figure 4b) show a broad diffraction peak at $2\theta = 22^\circ$, attributed to amorphous silica [50]. Moreover, the XRD patterns of the Ce-modified catalysts show no characteristic reflections associated with crystalline cerium oxide phases. This suggests that Ce species were successfully incorporated into the SBA-15 framework, rather than forming segregated CeO_2 domains. If any cerium-containing phases are present outside the mesostructure, they are likely amorphous or exist as highly dispersed clusters or particles below the detection limit of X-ray diffraction [51].

The nanostructure of the 0.1Ce/SBA-15 and 0.2Ce/SBA-15 catalysts was studied by transmission electron microscopy. As shown in Figure 5, both materials show the typical hexagonal, unidirectional pore arrangement, in agreement with the low-angle XRD patterns. The pore diameter estimated from the images is ~ 6 nm, while the wall thickness is about 2 nm. Using this technique, it was possible to observe small areas of high contrast in both samples (Figures 5b, 5d), which could correspond to small amorphous or highly dispersed crystalline domains of cerium oxides segregated outside the amorphous silica network.

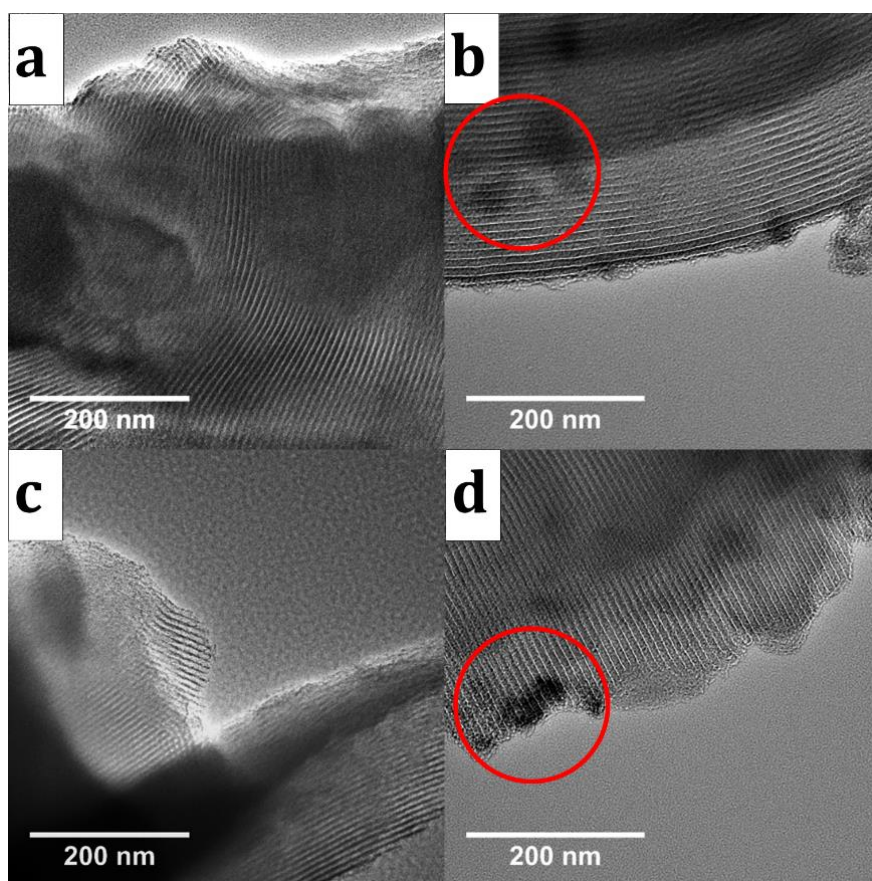


Figure 5 TEM images of: 0.2Ce/SBA-15 (a and b), and 0.1Ce/SBA-15 (c and d).

The morphology of the synthesized materials was studied by scanning electron microscopy. SEM images of the catalysts (Figure 6) show the typical form of SBA-15 agglomerates with rod-like macrostructures, composed of small, interconnected wheat grain domains with relatively uniform sizes of ~ 1.4 μm wide and 2-3 μm high. Since the surface of those aggregates is glossy and regular, the formation of organized structures can be inferred [51, 52].

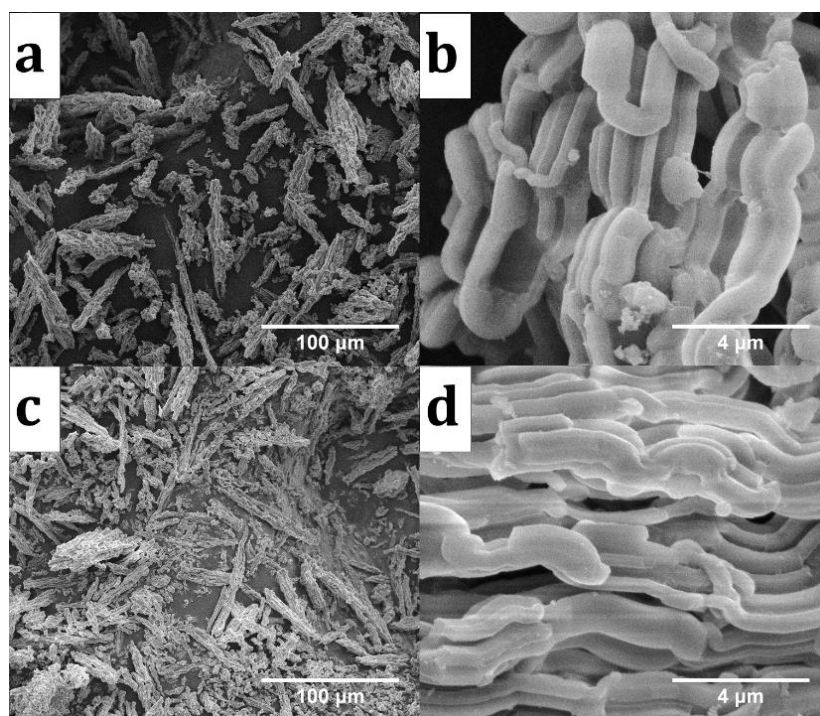


Figure 6 SEM images of 0.2Ce/SBA-15 (a and b), and 0.1Ce/SBA-15 (c and d) at different magnifications.

Nitrogen adsorption-desorption isotherms for SBA-15 and both Ce-incorporated SBA-15 catalysts are shown in Figure 7. All the samples exhibit isotherms of type IV with an H1 hysteresis loop, according to the IUPAC classification, typical of mesoporous structures [53], as evidenced by low-angle X-ray diffraction (Figure 4a). The well-defined step at a relative pressure of 0.65 is characteristic of capillary condensation within the mesopores. The hysteresis loop has two branches that are almost vertical and nearly parallel, associated with a narrow pore-size distribution.

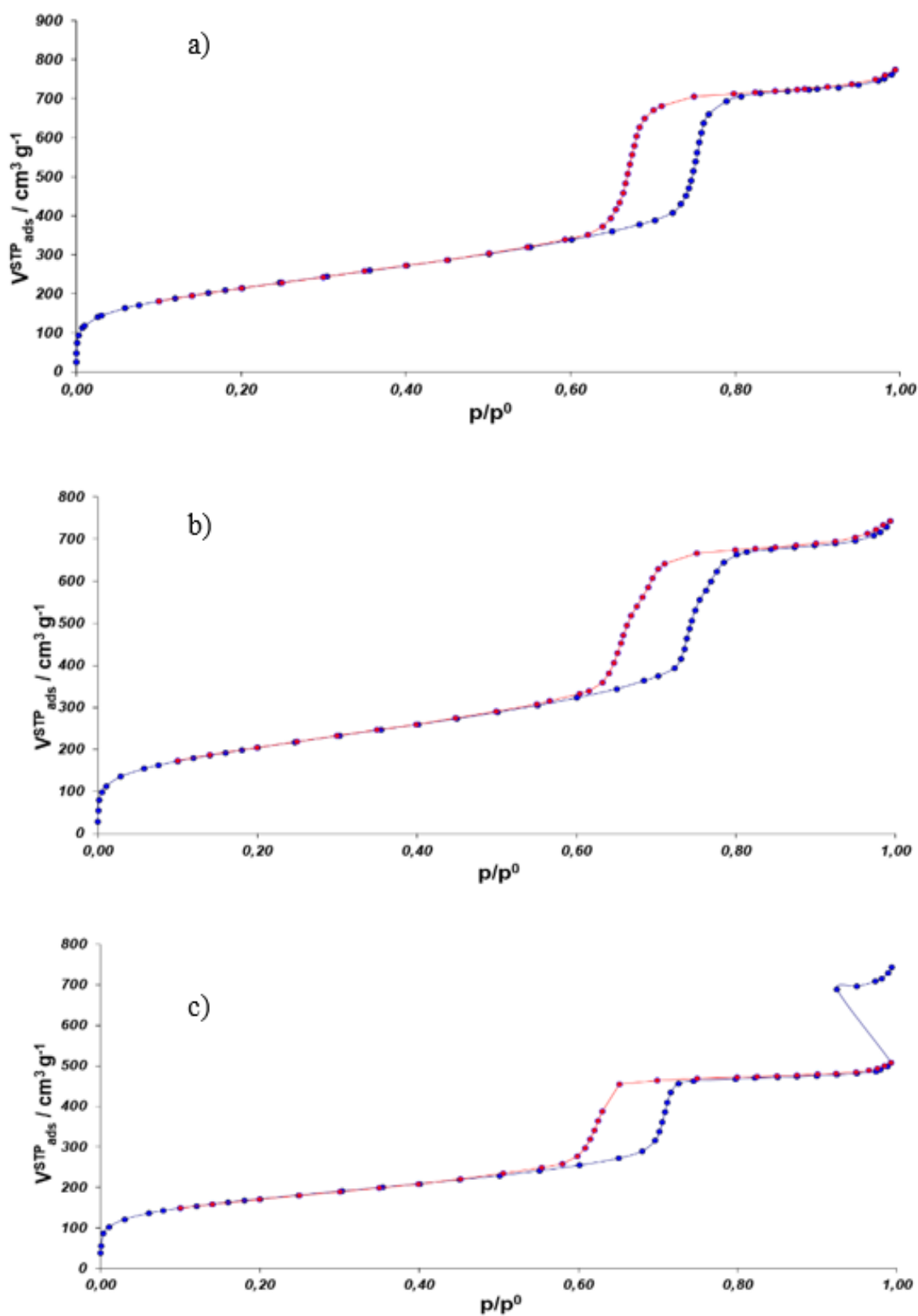


Figure 7 Nitrogen adsorption-desorption isotherms of (a) 0.2Ce/SBA-15, (b) 0.1Ce/SBA-15, and (c) SBA-15.

BET-specific surface, average pore diameter, and pore volume are presented in Table 3. Specific surface values of 0.2Ce-SBA-15 and 0.1Ce-SBA-15 increase with respect to pure SBA-15. Similarly, pore volume and pore size were significantly increased with increasing Ce content [36]. Timofeeva

et al. attribute the slightly increase of parameters such as pore diameter and wall thickness as the cerium content rises to the difference between the ionic radius of $\text{Ce}^{+3}/\text{Ce}^{+4}$ (1.01–0.87 Å) and Si^{+4} (0.39 Å), which causes a change in the cell parameter (a_0), and the Ce–O bond length (2.06 Å) [54] which is longer than that of Si–O (1.55 Å) [55]. Hence, the pore size and pore volume became larger.

FT-IR spectra of pure SBA-15 and the two Ce-incorporated-SBA-15 samples are shown in Figure 8. Owing to the material's hydrophilic character, a broad band at 3440 cm^{-1} (together with a band at 1630 cm^{-1} corresponding to adsorbed water), which masks the silanol stretching vibration band that should appear at 3750 cm^{-1} . Other bands at 1080, 960, 800, and 460 cm^{-1} are attributed to the asymmetric stretching vibration of tetrahedral Si–O, Si–OH bending, and Si–O–Si symmetric stretching and bending, respectively.

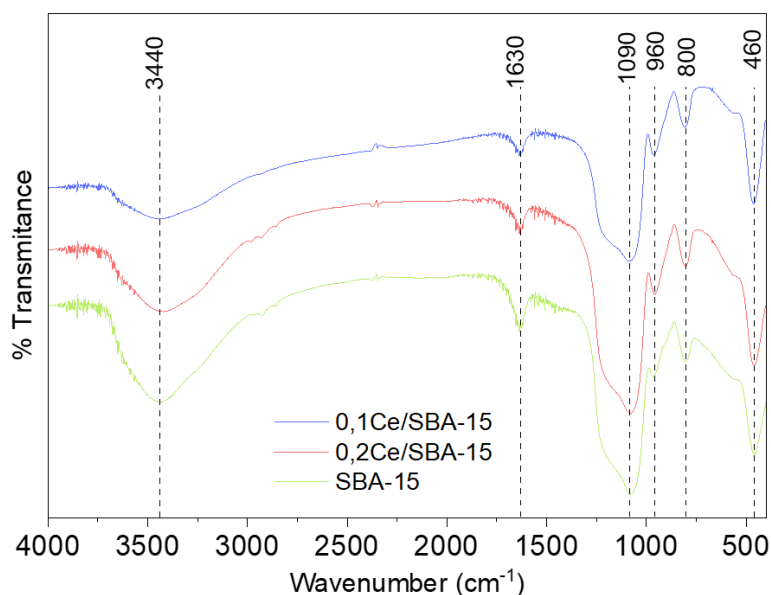


Figure 8 FT-IR spectra of pure SBA-15 and the synthesized materials (0.1Ce/SBA-15, 0.2Ce/SBA-15).

The acidic sites facilitate the breaking of C–C and C–O bonds in organic substrates and can then catalyze most reactions during the pyrolysis of lignocellulosic biomass [24]. IR spectroscopy of pyridine adsorbed on a solid is a powerful tool for identifying the nature of acid sites [56]. Applying this technique, IR spectra of the samples were obtained after pyridine desorption by evacuation at different temperatures following adsorption at room temperature, and characteristic IR bands were identified. Figure 9 presents the FT-IR spectra for the samples 0.1Ce/SBA-15 and 0.2Ce/SBA-15 after the adsorption of pyridine and successive heating at 100 and 150°C .

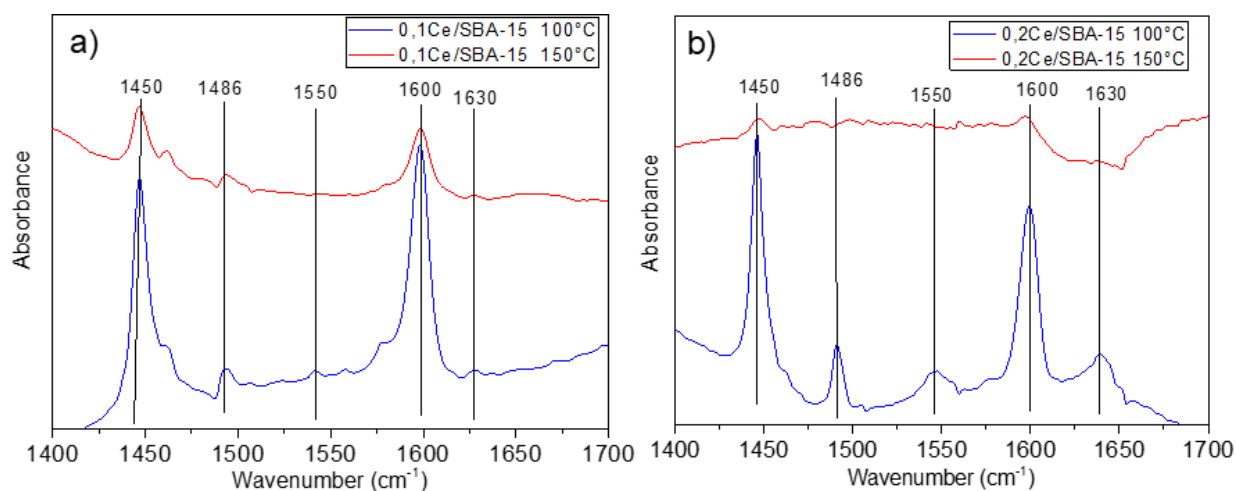


Figure 9 FT-IR spectra of a) 0.1Ce/SBA15 and b) 0.2Ce/SBA15, with pyridine desorbed at 100°C and 150°C.

Pyridine is known to form hydrogen bonds with silanol groups present in its structure, whose hydroxyls cannot protonate it. Therefore, all our samples present bands corresponding to silanol-bonded pyridine at 1600 and 1450 cm^{-1} [57], which disappear entirely after evacuation at 150°C for sample 0.2Ce/SBA-15 (Figure 9b). However, according to the literature and our previous reports [58, 59], the formation of a Lewis adduct with the adsorbed pyridine is identified by IR absorption bands at approximately 1600–1620 and 1445–1455 cm^{-1} . As can be seen in Figure 9, after evacuation at 100°C, the integrated absorbances of the bands at 1600 and 1450 are slightly higher for the 0.1Ce/SBA-15 sample (Figure 9a) than for the 0.2Ce/SBA-15 material (Figure 9b); this could show a contribution (overlap of these bands) of the pyridine bound to the Lewis acid sites in 0.1Ce/SBA-15, which are slightly stronger in retaining pyridine molecules up to 150°C. On the other hand, the presence of a band at 1630 cm^{-1} , notably more intense in the 0.2Ce/SBA-15 sample, can be attributed to the interaction of pyridine with the Brønsted acid hydroxyls [60]. In this sense, Parvulescu et al. [33] assigned this band to pyridine adsorbed on hydroxyl ions. Under evacuation at 150°C, the band at 1632 cm^{-1} tends to disappear, indicating that these Brønsted sites are of very weak character. Notably, although sample 0.1Ce/SBA-15 possesses silanol nests, these present a very low intensity for the 1632 cm^{-1} band. Therefore, it is possible to suggest that the acidic properties of these silanol nests are likely modified by the inductive effect of Ce present in the structure. Thus, changes in the electron density around Si due to charge imbalance, differences in electronegativity, or local structural deformation resulting from the introduction of Ce in the vicinity of hydroxyl-bearing silicon can weaken the Si–OH bond in the silanol nests, increasing their acidic strength.

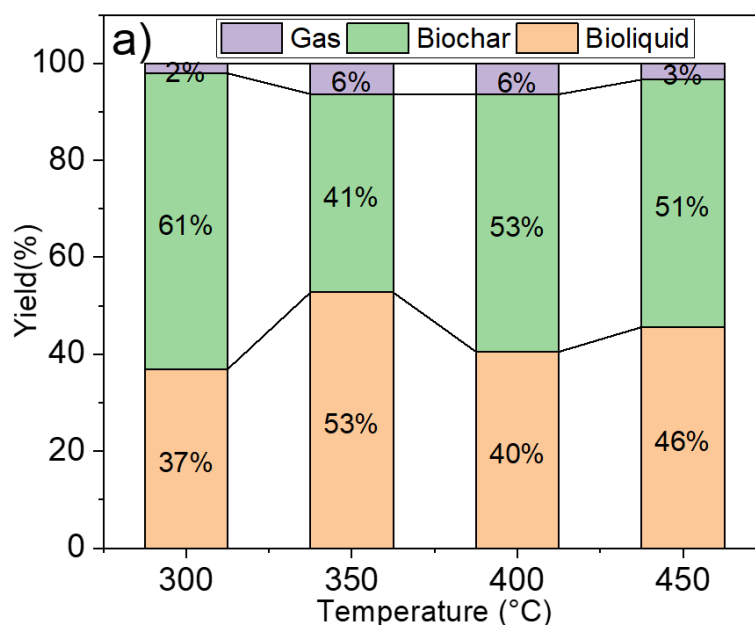
In general, all catalysts exhibited similar spectra, indicating that the synthesized catalysts (0.1Ce/SBA15 and 0.2Ce/SBA15) presented bands associated with both sites, B and L. To estimate which type of acid sites were most abundant, the L/B and L/(L + B) ratios at 100 and 150°C were calculated. The results shown in Table 4 indicate that both Ce modified catalysts have more Lewis acid sites than Brønsted acid sites. Then, 0.1Ce/SBA15 shows the highest proportion of L sites, which could be attributed to the high dispersion of metal species achieved on the surface. Regarding the strength of the sites, the desorption of pyridine at 150°C indicated that the Lewis acid sites are stronger than the Brønsted acid sites.

Table 4 Area of FT IR peaks associated with acid sites after pyridine desorption at 100°C and 150°C.

	L (u.a)	B (u.a)	L/B	L/(L + B)	T (°C)
0.2Ce/SBA-15	3.05	0.80	3.81	0.79	100
0.1Ce/SBA-15	1.81	0.02	90.50	0.99	
0.2Ce/SBA-15	0.030	0.04	7.50	0.88	
0.1Ce/SBA-15	0.41	0.01	41.00	0.98	150

3.3 Impact of Temperatures on Output Yield

Non-catalytic pyrolysis experiments were conducted at four temperatures (300, 350, 400, and 450°C) to evaluate the influence of temperature on bio-oil yield (Figure 10). The results show that increasing the reaction temperature led to a decline in liquid yield, with the maximum bio-oil production observed at 350°C. This temperature was therefore identified as the optimal condition for the pyrolysis of pear pulp waste under the studied parameters.

**Figure 10** Yields (%) of the non-catalytic pyrolysis products (PP waste 3 g) at different temperatures.

The influence of temperature on the product family's selectivity (Figure 11) and specifically on sugar compounds' selectivity (Figure 12) is shown below.

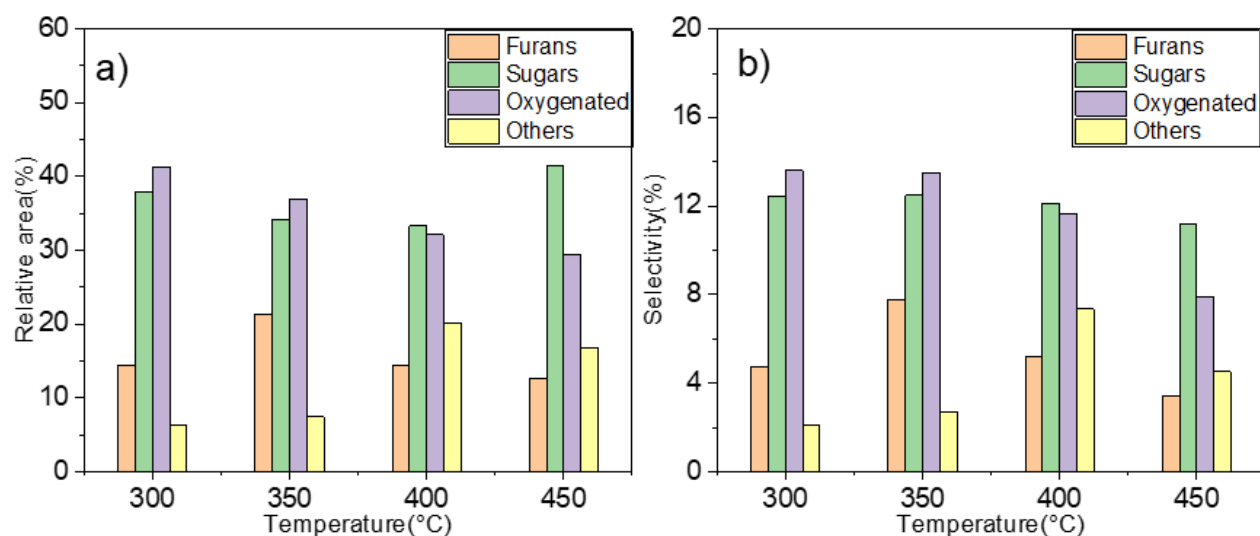


Figure 11 Product families for non-catalytic fast pyrolysis of PP waste at different temperatures: (a) Relative area and (b) Selectivity (%).

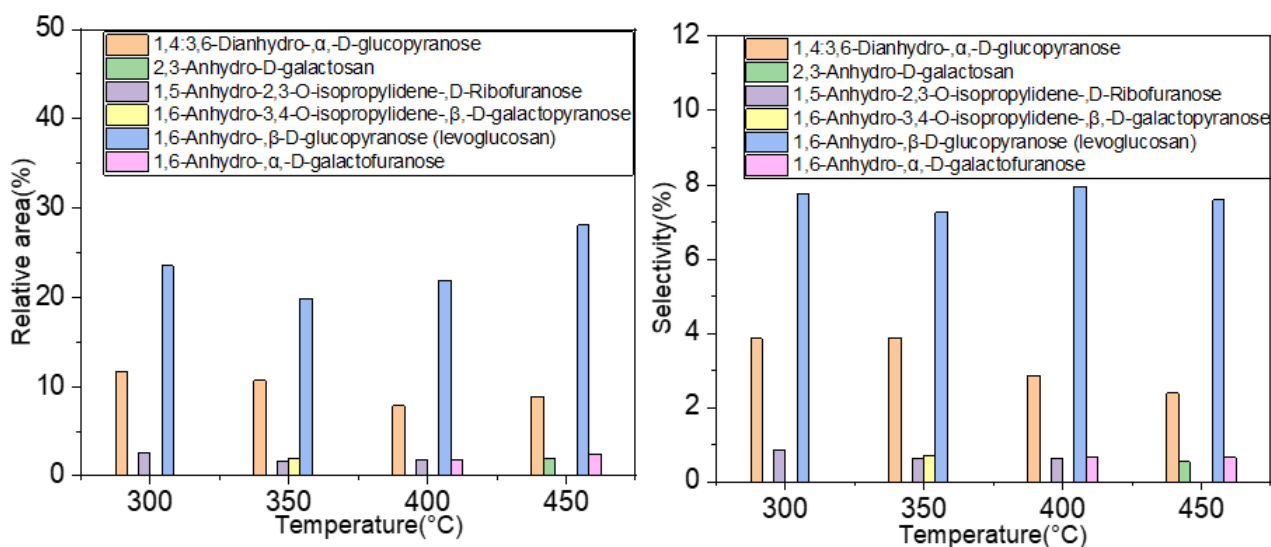


Figure 12 Sugars (a) Relative area and (b) Selectivity for non-catalytic fast pyrolysis of PP waste, obtained at different temperatures.

3.4 Chemical Compositions of Bio-Oil at Optimum Temperature

To examine the catalytic effect on bio-oil yield, the PP pyrolysis reaction was carried out at the defined optimum temperature of 350 °C for 15 min using an auxiliary reactor. Figure 13 shows the yield of bio-oil, biochar, and gas for SBA-15, 0.1Ce/SBA-15, and 0.2Ce/SBA-15, concluding that pyrolysis with the 0.1Ce/SBA-15 catalyst presents a higher yield of bio-oil compared to the other materials.

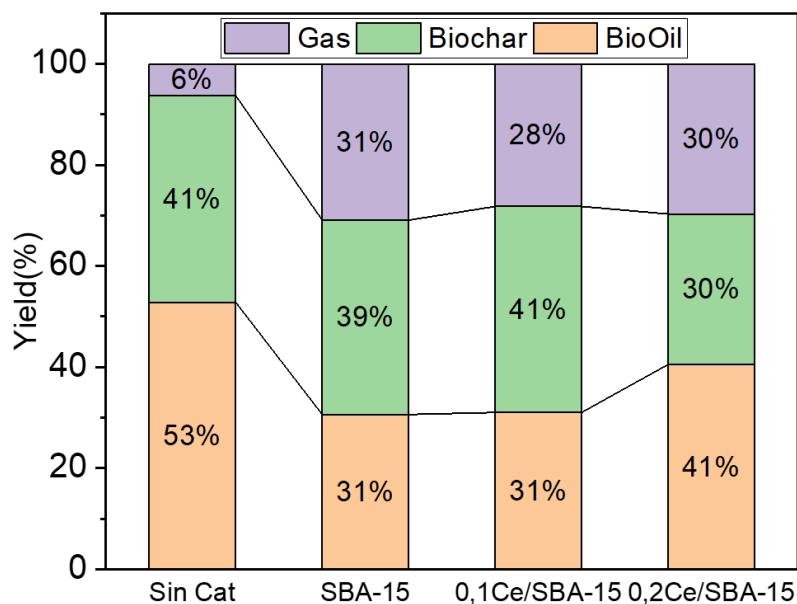


Figure 13 Yields (%) of the pyrolysis catalytic products (PP waste 3 g) at T = 350°C.

The compounds identified in the bioliquid were categorized into four groups: furans, phenols, sugars, oxygenated compounds, and others. Compounds were identified using the National Institute of Standards and Technology (NIST) mass spectral library. Compounds that appeared consistently with high probability were selected and quantified. The yield (% by weight with respect to the initial amount of PP waste) and the selectivity of the main products obtained is given in Figure 14a and Figure 14b.

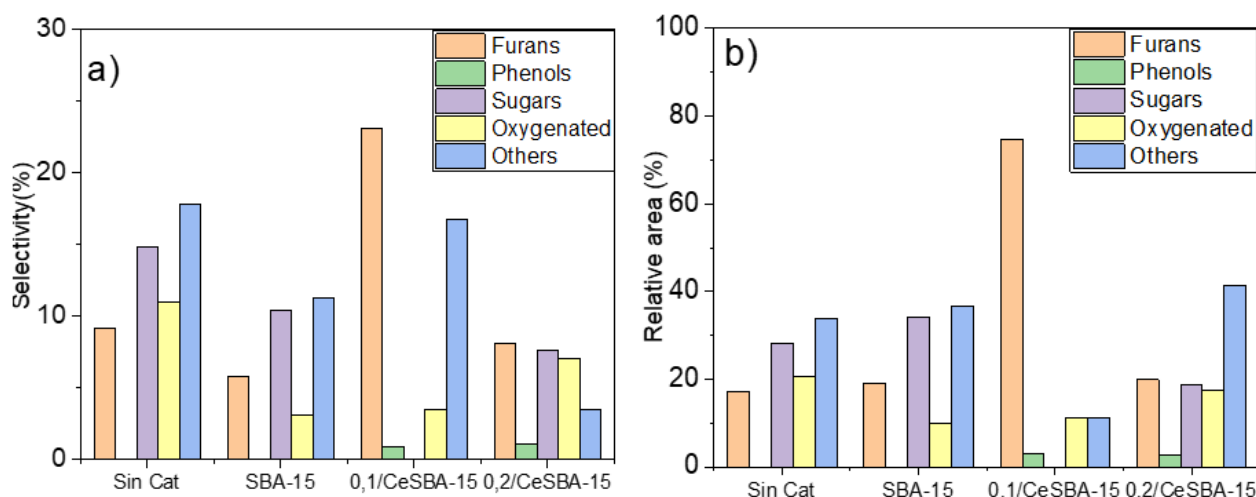


Figure 14 Product families for non-catalytic and catalytic fast pyrolysis of PP waste. (a) Selectivity (%) and (b) Relative area. Reaction conditions: T = 350°C, biomass 3 g, catalyst to sample weight ratio 10, catalyst: (1) No cat, (2) SBA-15, (3) 0.1Ce/SBA-15, (4) 0.2Ce/SBA-15.

The selectivity of furans in pyrolysis with 0.1Ce/SBA15 catalyst produced Furans (23.15%), while with 0.2Ce/SBA15 catalyst, produced (18.38%) (Figure 14a). It should be noted that other by-products were not considered due to their very low value.

Figure 14b shows the relative area of the different product groups from the pyrolysis of PP waste with and without a catalyst. In the case of catalytic pyrolysis over SBA-15 and 0.2Ce/SBA-15, mainly sugars and furans were significantly affected by the catalyst, with their fractions present in the bio-oil. Since SBA-15 only presents terminal silanol groups and lacks Brønsted and Lewis acid sites, the conversion efficiency of the pyrolytic vapors produced during pyrolysis might be low. On the contrary, with 0.1Ce/SBA-15, the formation of oxygenates, phenols, and sugars decreased, and that of furans increased. These results indicate that strong Lewis acid sites are responsible for the formation of furanic compounds.

As can be seen (Figure 15), the experiments performed with and without a catalyst produced a wide variety of furans in the liquid (including furfural and 5-methyl-2(3H)-furanone, among many others), with a marked selectivity towards Furfural.

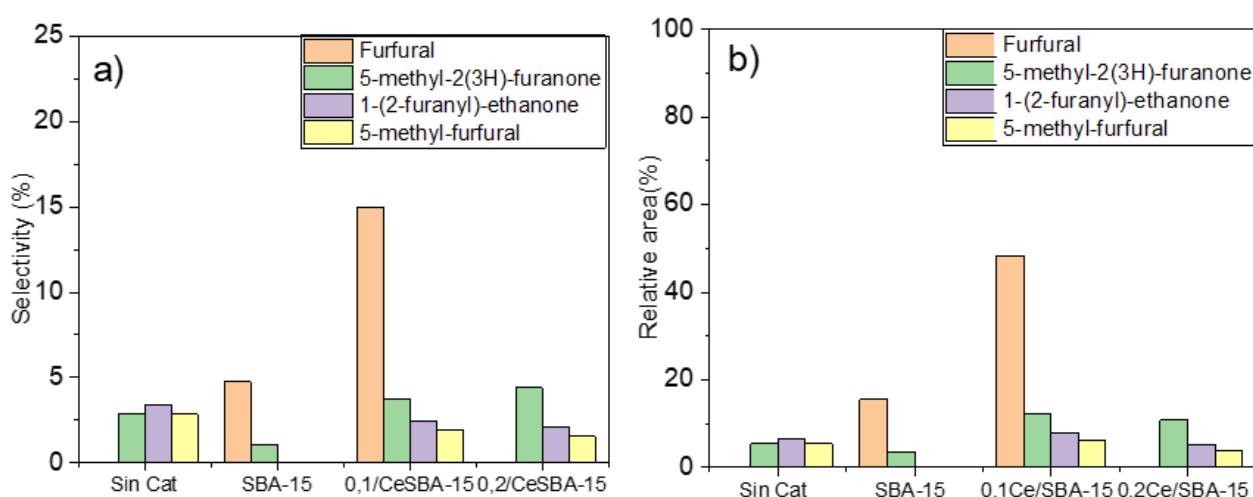


Figure 15 Furans (a) Selectivity and (b) Relative area for non-catalytic and catalytic fast pyrolysis of PP waste. Reaction conditions: $T = 350^{\circ}\text{C}$, biomass 3 g, catalyst to sample weight ratio 10, catalyst: (1) No cat, (2) SBA-15, (3) 0.1Ce/SBA-15, (4) 0.2Ce/SBA-15.

Comparing catalysts with different Ce contents, the lower furfural production with 0.2Ce/SBA-15 may be due to the uneven distribution of Ce, leading to the accumulation of excess Ce on some parts of the catalyst surface, which eventually forms larger particles and limits catalytic performance. This could be observed more clearly in Figure 5. The incorporation of Ce linked to the surface via oxygen in the silica framework would unfavorably affect the oligomerization process and the formation of Ce oxide clusters or small nanoparticles (not detected by XRD). This feature leads to Ce species being finely dispersed on the surface, which act as moderate L acid sites, improving the catalytic activity of 0.1Ce/SBA-15.

4. Conclusions

The fast pyrolysis of PP waste from candy and jam production was evaluated using a catalytic system to enhance both bio-liquid yield and quality. The catalyst enhanced both selectivity and the relative area of key compounds, particularly furfural at 350°C , with 0.1Ce/SBA-15 achieving 15% selectivity and a 48% relative area. These results demonstrate the practical potential of pear pulp waste as an inexpensive, abundant feedstock for producing valuable platform chemicals, such as furfural.

Future work should assess the effect of varying catalyst amounts, both lower and higher than 10%, to determine whether selectivity or catalytic performance can be further improved. Additional studies focused on catalyst stability, regeneration, and the evaluation of other agro-industrial residues would help clarify the feasibility of scaling this process for industrial applications.

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Author Contributions

Orlando N. Guiñazú, Edgar M. Sánchez Faba: Investigation, Formal analysis, Visualization, Writing – Original Draft; Griselda A. Eimer, Laura E. Moyano: Conceptualization, Formal analysis; Horacio Falcón: Funding acquisition, Writing – Review & Editing.

Competing Interests

The authors have declared that no competing interests exist.

AI-Assisted Technologies Statement

Artificial intelligence (AI) tools were used solely for basic grammar correction and language refinement in the preparation of this manuscript. Specifically, Google AI was employed to improve the readability and linguistic clarity of the English text. All scientific content, data interpretation, and conclusions were developed independently by the author. The authors have thoroughly reviewed and edited the AI-assisted text to ensure its accuracy and accept full responsibility for the content of the manuscript.

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