

Chapter 5.14. Optimal slow pyrolysis of pomace reaction conditions for the generation of a feedstock gas for hydrogen production

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ABSTRACT

The present research is aimed to explore optimal reaction conditions for the generation of a gas product, through the slow pyrolysis of pomace, to be used as a feedstock for the production of H₂ by the absorption enhanced reforming of methane (AER) process scheme. Pyrolysis experiments were performed in a tubular reactor under conditions of rich syngas production (300–450 °C) and for specific solid heating rates (5-20 °C/min). Permanent gases (CO, CH₄ and CO₂), aromatic tars, and chars were quantified at different heating rates and isothermal conditions. Results indicate that at a temperature of 400 °C a maximum of 71.5% W of non-condensable volatile matter (NCVM) can be obtained along with 25.4% W of condensable volatile matter (CVM), while only 3% W of residual matter (RM) was produced. At this favorable conditions (NCVM) a gas composition of 41% CO, 38% CO₂ and 21% CH₄ was generated. A thermodynamic analysis of this product gas was performed under the steam reforming of methane (SMR) combined with CO₂ solid absorption by CaO for the production of high purity H₂. Thermodynamic calculations using a steam to methane ratio (S/C) of 4 and 1 mol of CaO/mol of CH₄ indicate that a maximum H₂ production is achieved at 690 °C along with a composition of 74.1 % H₂, 16.2% CO, 7.6% CO₂ and 2.1% CH₄ with no carbon formation.

Keywords: Pyrolysis of Pomace, Hydrogen production and CO₂ capture

1. Introduction

The use of biomass for the production of energy has become an important strategy aimed to reduce the harmful environmental impact of today's use of fossil fuels. Energy derived from biomass and solid wastes, has been the heart of many thermochemical and biochemical processes [1]. In thermochemical processes, gasification and pyrolysis has reached greater energy conversions than direct combustion. During gasification, fuel molecules (H₂, CH₄, CO and CO₂) are produced, while biomass is transformed into gaseous

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products [2]. Furthermore, biofuels produced from various lignocellulosic materials such as wood, agricultural or forest residues, have the potential to be a valuable substitute (or supplement to gasoline biofuels) to liquid or gaseous fuels for the transport sector [3]. One specific example of a biomass waste is the apple pomace, which is the residue generated in the process of extraction of apple juice. This apple pomace, is formed by a complex mixture of shell, seed kernel, calyx, stem and soft tissue, which is representative of the pomace, and this contains mainly cellulose, hemicellulose, lignin and pectin.

Moreover, a great deal of research has been made focused on flash and vacuum pyrolysis, process conditions and its yield. However, slow pyrolysis has been overlooked, probably because in this kind of processes solid residues are the interested product and condensable and non-condensable products are burned or directly emitted to the atmosphere [4] and as a consequence, up to date there are no studies involving the slow pyrolysis of Mexico's apple pomace. This condensable and non-condensable matter that usually is called bio-oil and bio-gas, respectively; is suitable to be employed as feedstock for hydrogen production [5].

Two main technologies for hydrogen production that have been explored in recent years are biomass steam gasification and biomass slow pyrolysis [6]. It has been reported that fast pyrolysis followed by steam methane reforming of oil delivers less H_2 yield. The H_2 production cost from supercritical water gasification is too high. Compared with fast pyrolysis and supercritical water gasification, steam gasification performs better. However, the efficiency of the steam methane reforming of the products from the slow pyrolysis process can be enhanced towards the production of hydrogen from biomass (apple pomace) [7].

Furthermore, in biomass-derived carbohydrates steam reforming reactions, if CO_2 is removed immediately, according to Le Chatelier principle, its partial pressure decreases and the equilibrium shifts to the direction that favors hydrogen production [8-11]. This makes possible high fuel (i.e. CH_4) conversion at relatively low temperatures and production of high quality hydrogen (95%) with only traces of carbon oxides (in the order of 1 vol. %). This concept has been called absorption enhanced steam reforming (AESR) [12]. Recent experiments by Yi and Harrison [13] demonstrated that in the presence of an absorbent fairly good conversion and very low concentrations of CO can be achieved even at very low pressures (1 bar) and temperatures (460 °C). A potential absorbent material should have good CO_2 capacity at high temperatures, be easily regenerable and thermally stable to allow a cyclic absorption-desorption process. Experimental studies have demonstrated that in situ carbonation of CaO within the steam reformer is able to generate high H_2 concentrations [9-10]. Thus, this absorbent (CaO) have become highly attractive to be used under the AESR process. Therefore, a thermodynamic analysis can be of paramount importance to investigate fundamental reaction parameters influencing the output of H_2 -rich gas from the AERS of non-condensable volatile matter (NCVM) produced from the slow pyrolysis of apple pomace. However, NCVM composition is of great importance, since from this depends the final hydrogen production purity and quantity.

Hence, the present research is aimed to explore optimal reaction conditions for the generation of a gas product (NCVM), through the slow pyrolysis of pomace, to be used as a feedstock for the production of H_2 by the absorption enhanced reforming of methane (AER)

process scheme. Furthermore, a thermodynamic analysis will be performed to explore the hydrogen production by the AER of the NCVM.

2. Experimental

2.1 Sample Characterization

Apple pomace samples were collected from the northern state of Chihuahua (Cauhtémoc, Chihuahua) and subjected to a drying process, crushed, grounded and sieved to achieve a particle size of 150 μm . The elemental and proximal analyzes for the apple pomace sample were performed using a Carlo Erba EA-1110 elemental analyzer and an atomic emission spectrometer coupled with ICP (ICP Thermo Jarrell Ash IRIS/AP DUO). Lignin, cellulose, hemicellulose and pectin content from the pomace were determined using gravimetric techniques, described in ASTM (E 1756-95, D1106-95) and ASTM (D1103-60). Moisture, volatiles and ash content was determined according to the procedure described in ASTM E (871-82), ASTM (872-82) and ASTM (1755-1795), respectively. In order to determine the particle size (dp), samples were analyzed with dimensions: dp < 150 μm (150 μm), 150 < dp < 180 μm (180 μm), 180 < dp < 250 μm (250 μm), and 250 < dp < 450 μm (450 μm)

2.2. Determination of bagasse pyrolysis parameters

Operating parameters that directly influence biomass pyrolysis are: temperature, particle size (dp), heating rate and residence time [14]. The production of a greater amount of volatile matter was the criterion applied for determining the value of the operating parameters. For this purpose, Thermogravimetric Analysis (TGA) tests were performed in TA Instruments Q500. TGA conditions employed to find optimal values for temperature and particle size were; a N_2 flowrate of 150 cm^3/min and a heating rate of 10 $^\circ\text{C}/\text{min}$. In order to verify the effect of the heating rate over the production of volatile matter, three samples of cane bagasse with the same particle size were exposed at different heating rates; 5, 10, 15 and 20 $^\circ\text{C}/\text{min}$. Residence time, which was defined as the time required for a 90% volatilization of the sample, was determined by varying the residence time at different temperatures (pyrolysis in stages) [15]. This was achieved by the combination of the dynamic and isothermal TGA operating modes under the following conditions: particle size of 420 < dp < 840 μm , heating rate of 10 $^\circ\text{C}/\text{min}$, N_2 flowrate of 100 cm^3/min , isotherms of 150, 200, 250, 300, 350, 400, 450, 500 and 550 $^\circ\text{C}$ and residence time of 60 min.

2.3. Quantification of the pyrolysis products

The carbon residue percentage (CR) was obtained from the fractioned pyrolysis performed in the TGA using a residence time of 60 min. The condensable matter was gravimetrically quantified during the fractioned pyrolysis and this was performed in a fixed-bed tubular reactor coupled to a condensing system as described in Figure 1.

The pyrolysis apparatus was composed of a stainless steel tubular reactor, heated by an electrical furnace to temperatures up to 600 $^\circ\text{C}$ in an inert N_2 atmosphere (see Fig. 1). The stainless steel tubular reactor had an internal diameter of 2 cm and a heated length of 27

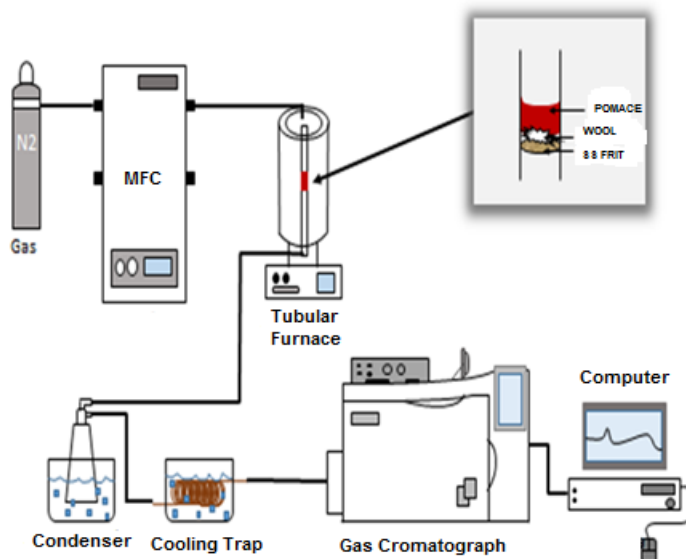


Fig. 1. Experimental reactor setup for the pyrolysis of pomace.

cm. Apple pomace samples were introduced into the center of the preheated reactor and held with a porous stainless steel frit and quartz wool. The carrier gas (N_2) was also used as a tracer. Its flow rate ($100 \text{ cm}^3/\text{min}$) was controlled by a mass flow rate regulator (Brooks Instrument). Condensable volatiles are trapped in a copper coil condenser cooled to 2°C . In order to determine the amount of mass of condensable volatiles, the weight change before and after each cycle of the pyrolysis was measured. The volatile compounds were separated and identified by gas chromatography using a Perkin Elmer Clarus 500. Detection of non-condensable gases (N_2 , H_2 , CO , CO_2 , CH_4 , C_2H_4 , and C_2H_6) was performed simultaneously with the devolatilization process. Condensable products were collected and stored for each isotherm to be analyzed later. In order to determine the amount of condensable volatile products, the heat exchanger was weighted before and after each pyrolysis stage using an analytic balance. The condensable volatile matter percentage (% CVM) for each isotherm was calculated according to the following expressions:

$$\text{CVM} = \text{CVM}/\text{MB}_1 \times 100 \quad (1)$$

where: MB_1 is the initial pomace mass, the percentage of non-condensable volatile matter (%NCVM) was obtained from the pyrolysis material balance:

$$\% \text{NCVM} = 100\% - \% \text{CVM} - \% \text{RC} \quad (2)$$

where the % RC is the percentage of carbon residue.

2.4. Thermodynamic analysis

All calculations were performed through the use of the equilibrium module of the HSC chemistry software [16]. HSC calculates the equilibrium composition of all possible combination of reactions that are able to take place within the thermodynamic system. These equilibrium calculations make use of the equilibrium composition module of the HSC program that is based on the Gibbs free energy minimization technique. The GIBBS program of this module finds the most stable phase combination and seeks the phase compositions where the Gibbs free energy of the system reaches its minimum at a fixed mass balance, constant pressure and temperature.

3. Results and discussion

3.1 Physicochemical characterization of pomace

Table 1 presents results of the physicochemical characterization performed on the apple pomace. These results are comparable to the ones reported in the literature related to other typical bagasse biomass [17-26].

The thermal decomposition of the apple pomace reveals two main regions attributed to the decomposition of cellulose and hemicellulose. The first decomposition mass loss that occurs at low temperatures can be associated to the process of pyrolysis of hemicellulose and at higher temperatures the weight loss is associated with decomposition of cellulose. Moreover, mass losses for the decomposition of lignin are not observed in this temperature range. Understanding of the volatilization of apple pomace is important because pyrolysis is the first step in a process of gasification or combustion [27]. Furthermore, in this TGA plot it is observed that the greatest amount of volatile material is produced at a heating rate of 5 °C/min, which is the curve that ends its decomposition at a lower temperature (~540 °C). When heating rate increases, the required time to reach a certain temperature value increases, enabling dehydration, depolymerization, carbonylation, carboxylation and transglycosylation reactions. As a consequence, the amount of devolatilized matter is increased. The obtained curves at different heating rates, after a certain decomposition stage at high heating rate, reach a common value typical of the mass solid residue [28]. The largest sample weight loss is located from a temperature range of 200-450 °C, as can be observed in Fig. 1, and this can be attributed to the devolatilization process. Analyzing this behavior, according to the literature the weight loss that occurs at 200 °C is related to the beginning of the lignin and hemicellulose pyrolysis contained in the apple pomace [29]. From 250 and up to approximately 350 °C the high decomposition rate arises and in this region the maximum devolatilization of hemicellulose, cellulose and lignin is achieved. Remaining molecules of these compounds generate the next weight loss that corresponds to the temperature (up to 550 °C) in which the reaction ends.

Table 1. Apple pomace physicochemical data.

Parameter	Units	Magnitude
Elemental analysis^a		
C	%	47.98
H	%	6.65
N	%	0.78
S	%	-----
O	%	37.5
Composition analysis^{a,b}		
Cellulose	%	47.5
Hemicellulose	%	27.8
Lignin	%	22.4
Proximal analysis		
Humidity ^c	%	8.87
Volatile matter	%	81.32
Fixed carbon	%	6.41
Ash (420 < d _p < 840 μm)	%	5.10
Ash composition		
Al	%	0.51
B	%	11.43
Ba	%	37.75
Ca	%	2.01
Cr	%	0.80
Cu	%	8.50
Fe	%	0.35
K	%	7.73
Mg	%	1.54
Mn	%	10.24
Na	%	0.23
Ti	%	17.51
V	%	0.56
Extractable		
Ethanol	%	2.69
Physical properties		
Density	Kg/m ³	110.3
Calorific value	KJ/Kg	7639.18

a Dry bagasse basis **b** Extractable free **c** Wet basis

Experimental TGA curves obtained for the apple pomace under different heating rates are presented in Figure 2. Figure 3 presents a TGA plot of the apple pomace biomass (APB) subjected to different particle size fractions at a heating rate of 10 °C/min. In this Figure it can be observed that particle sizes smaller or equal than 150 μm are the ones that allow the generation of the greater amount of volatile matter. The difference in volatiles production with respect to the size of the particles is attributed to a main reason: particles greater than 425 μm, during the devolatilization process, some problems arise related to the heat and mass transfer [30], as reported by Lou and Stanmore [31].

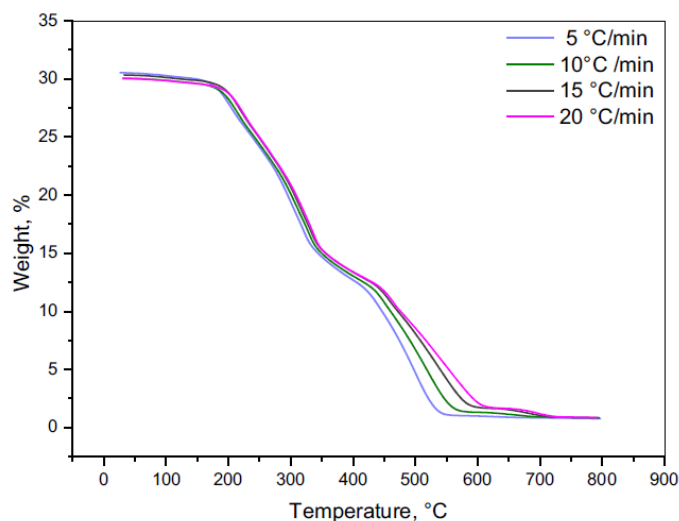


Fig. 2. TGA curves for the pyrolysis of apple pomace under N₂ at different heating rates.

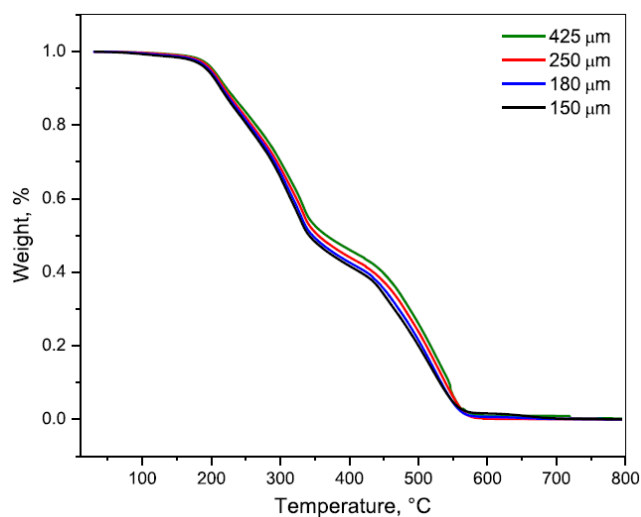


Fig. 3. TGA curves for the pyrolysis of the apple pomace under N₂ at a heating rate of 10 °C/min and different particle sizes.

3.2. Apple pomace pyrolysis fractions yield

Table 2 presents the percentage of each one of the product phases of the apple pomace pyrolysis obtained in each isotherm (condensable and non-condensable products and carbonaceous residue). In general, pomace pyrolysis studies that report the yield in each of its fractions were performed exclusively at the maximum working temperature (600 °C).

It can be observed (Table 2) that experimental values at a temperature of 550 °C are very close to the ones reported in other studies [17, 26]. Furthermore, it can be observed that at temperature of 400 °C the amount of the non-condensable volatile matter (NCVM)

is the greatest. Therefore, this is the temperature that will be selected for the further conversion to hydrogen through the absorption enhanced reforming of methane (AER) process scheme.

Table 2. Production of carbon, condensable and non-condensable matter for the pomace pyrolysis at several isotherms.

Temperature (° C)	CVM (%)	NCVM (%)	RC (%)
150	9.99	0.15	89.85
200	17.98	4.39	77.62
250	24.98	23.69	51.32
300	34.92	37.23	27.85
350	34.65	49.86	15.48
400	25.42	71.51	3.07
450	72.01	24.54	3.36
500	74.60	22.59	2.81
550	75.01	23.56	1.43
600	82.33	16.45	1.22

In this Table it can be observed that experimental values at a temperature of 550 °C are very close to the ones reported in other studies [17, 26].

Furthermore, it can be observed that at temperature of 400 °C the amount of the non-condensable volatile matter (NCVM) is the greatest. Therefore, this is the temperature that will be selected for the further conversion to hydrogen through the absorption enhanced reforming of methane (AER) process scheme.

Figure 5 presents the time response of the main gas composition from the pomace pyrolysis isotherm at 400 °C. Results indicate that at a temperature of 400 °C a maximum of 71.5% W of non-condensable volatile matter (NCVM) can be obtained along with 25.4% W of condensable volatile matter (CVM), while only 3% W of residual matter (RM) was produced. A Figure 3 shows that at this favorable conditions (NCVM) an average gas composition of 41% CO, 38% CO₂ and 21% CH₄ was generated.

3.3 Thermodynamic analysis

Considering the results obtained from the identification of most significant volatile compounds, in which an average gas composition of 41% CO, 38% CO₂ and 21% CH₄ was generated. This composition was used to perform a thermodynamic analysis under the AER process scheme and the high purity hydrogen can be produced through the combination of the following reactions:

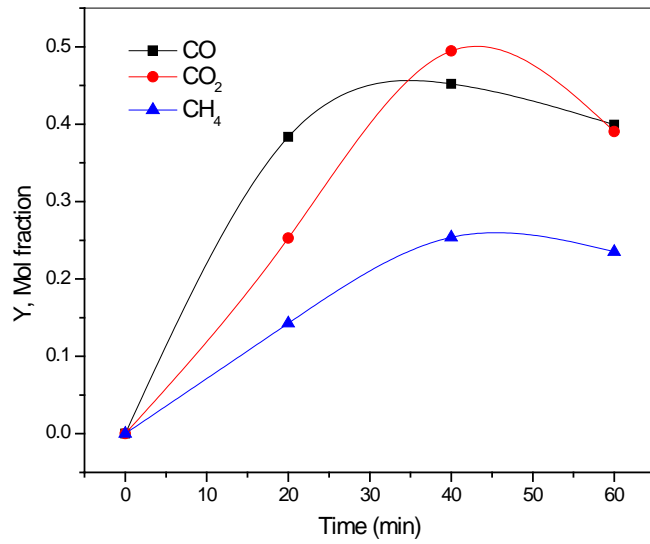


Figure 5. Reactor gas product composition at 400°C for the pomace pyrolysis

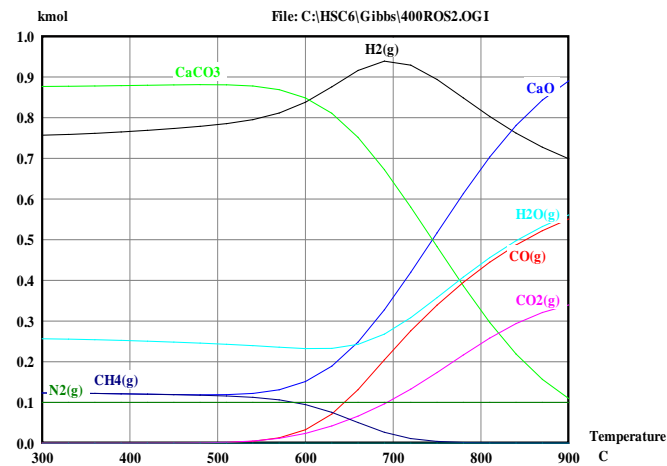


Figure 6. Thermodynamic analysis results for the gas product composition at 400°C for the pomace pyrolysis.

The AER is composed of reactions (3)-(5), which correspond to the steam reforming reactions in combination with the CO₂ absorption reaction by CaO (5) to produce high purity hydrogen. For thermodynamic modeling purposes the feed consisted of 1 kmol of the methane mixture with 4 moles of H₂O, which corresponds to a steam to carbon ratio of 4 (S/C) and 1 mol of CaO with results of this thermodynamic analysis presented in Figure 6.

According to results from Figure 6 the thermodynamic calculations using a steam to methane ratio (S/C) of 4 and 1 mol of CaO/mol of CH₄ indicate that a maximum H₂ production is achieved at 690 °C along with a composition of 74.1 % H₂, 16.2% CO, 7.6% CO₂ and 2.1% CH₄ with no carbon formation.

4. Conclusion and recommendation

The present research explored optimal reaction conditions for the generation of a gas product, through the slow pyrolysis of pomace, to be used as a feedstock for the production of H₂ by the absorption enhanced reforming of methane (AER) process scheme. Pyrolysis experiments were performed in a tubular reactor under conditions of rich syngas production (300–450 °C) and for specific solid heating rates (5-20 °C/min). Permanent gases (CO, CH₄ and CO₂), aromatic tars, and chars were quantified at different heating rates and isothermal conditions. Results indicate that at a temperature of 400 °C a maximum of 71.5% W of non-condensable volatile matter (NCVM) were obtained along with 25.4% W of condensable volatile matter (CVM), while only 3% W of residual matter (RM) was produced. At this favorable conditions (NCVM) a gas composition of 41% CO, 38% CO₂ and 21% CH₄ was generated. A thermodynamic analysis of this product gas was performed under the steam reforming of methane (SMR) combined with CO₂ solid absorption by CaO for the production of high purity H₂. Thermodynamic calculations using a steam to methane ratio (S/C) of 4 and 1 mol of CaO/mol of CH₄ indicate that a maximum H₂ production is achieved at 690 °C along with a composition of 74.1 % H₂, 16.2% CO, 7.6% CO₂ and 2.1% CH₄ with no carbon formation.

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