Waste and Biomass Valorization

STRATEGY FOR THE DESIGN OF WASTE TO ENERGY PROCESSES BASED ON PHYSICOCHEMICAL CHARACTERISATION

--Manuscript Draft--

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Abstract:	Energy recovery from wastes is needed for cost-effective and sustainable management. For a given waste, the definition of suitable thermochemical conversion process schemes relies on devising a strategy based on several variables among which feedstock characterization is crucial. Depending on the properties of the fuel, the available waste resource may not be suitable for a specific application, for technical and sometimes for environmental reasons [1]. Within this framework, agro-industrial wastes (grape stem, beer bagasse and orange juice residues) were characterized and the results are used to design a strategy for their effective integration in waste-to-energy processes. Energy content, proximate and ultimate analysis, composition, ash fusibility and thermal behaviour were determined. For the physicochemical analysis UNE standard methods were used. Characterization results showed that the three wastes have good quality for thermochemical conversion with energy contents between 19 MJ/kg (beer bagasse) and 16 MJ/kg (orange juice residue) and ash contents below 10 % in all cases. However, some drawbacks were found: high moisture (76%), nitrogen (3.5%) and sulphur (0.2%) content for beer bagasse; elevated nitrogen (1.1%) and sulphur (0.15%) concentration for grape stem and nitrogen (1%) content for orange juice residue. All this information has been used to design a smart strategy for selecting a sustainable and environmental friendly waste to energy processes as part of a circular economy approach.
Response to Reviewers:	Response to the Reviewers' Comments Journal:Waste and Biomass Valorization

Article Ref:WAVE-D-18-01045
Title:Strategy for the design of waste to energy processes based on physicochemical characterisation

COMMENTS FOR THE AUTHOR:

Reviewer #1: WAVE-D-18-01045
The paper offers a smart strategy for identifying a suitable thermochemical process for a waste, based on feedstock physicochemical characterisation. The topic appears interesting, but the proposed approach appears not holistic and lacking of some crucial issues. The organisation of the paper should be restructured, in order to better show the innovation of the proposed strategy and its application.

INTRODUCTION
- Most part of the introduction (for example the description of each type of the analysis or the equations 1-10), can be moved in other part of the paper or deleted. In the introduction part of a paper, the crucial points of the topic under analysis should be stated, an overview of state of art should be shown, and the structure of the paper together with the indication of its main objectives should be inserted. For example, are there proposed other "smart strategies" in the already published literature? What is the contribution of this paper in the field?
Response:
The introduction part has been modified taken into account the comments of the reviewer.
The main objectives of the work are in the paragraph: "In order to help in the selection of the most adequate technology for a specific type of waste the aim of this work is to design a smart strategy to be used in sustainable and environmental friendly waste to energy processes as part of a circular economy approach."

METHODOLOGY
- This part should begin with a general description of the smart strategy with the indication of its main steps and support information for each of this step (for example the indication of carried analyses or indexes utilised in each step). To this aim, a clear and schematic figure (but at same time rich of information) could be inserted.
Response:
Methodology section has been modified according to the reviewer's comments.

SECTION 2.4
- Line 43. The selection of a product of interest (hydrogen, electricity, biofuels) cannot be related only to the suitability of a feedstock for a thermochemical process, but also to the technical performances of the process itself. For example, the production of a biofuel (but also of a chemical or hydrogen) requires strict requirements in terms of syngas cleaning and conditioning. Specifically, tar content in the syngas obtained from gasification represents the main issue for the syngas utilisation in final devices able to produce electricity or high added products (fuels, chemicals etc.). How the proposed strategy takes into account tar content and its related issues?
Response:
The product of interest is an input in the strategy. Once selected, the strategy evaluate the suitability considering the process used and the cleaning requirements. In the case of tar cleaning the technology considered in the strategy is based in a scrubber but this aspect is not development in the present paper because it will be included in the second part of the strategy.

- Table 2. The table appears not representative of the main fuel specifications. For example, physical properties of the feedstock (e.g. size and form) could be inserted. Furthermore, not only the requirements of a reactor are crucial for the suitability of a feedstock to the gasification process, but also the conditions (authothermal or allothermal), together with the indication of type of the gasifying agent (oxygen? Air? Steam?.
Response:
Table 2 has been modified according to the reviewer's comments.

- Table 3. Contaminants content is not the only issue to take into account for the combustion process. The type of reactor is crucial also in this case. For example, a

	fluidized bed combustor shows most stringent requirements than those of a moving grate furnace. Please, also if the table aims to be "only an example", in the reviewer's opinion, it could be better structured (as already indicated also for the table 2) in order to clarify all types of specifications to be taken into account. The same should be done for pyrolysis. Response: Table 3 is only an example but more information has been added in other table according to reviewer's comments. - Line 21. How the smart strategy takes into account the environmental aspects? The emissions in the air, water and soil by means of a thermochemical process can be not related only to the conversion stage, but they are strictly dependent from the air pollution control systems. Furthermore, a discussion about the fate of solid residues from processes (based on their composition that strictly depend from waste composition) is totally lacking in the paper. Response: This paper is focus in the first part of the strategy which is related to the physicochemical properties of the waste so it does not include details of the second part. Authors are totally agree with the comments of the reviewer, but the discussion of this aspect will be included in future work where the second part of the strategy will be described. FIGURE 1 The figure appears not clear and self-understanding. Why a moisture content below 15% is indicated? Is this value different for different types of processes? Maybe, it could be more correct to refer to "pre-treatment" as "mechanical pre-treatment" in order to distinguish it from drying stage that can be also a pre-treatment. Response: Figure 1 has been modified according to the reviewer's comments. Regarding to moisture content, 15% is indicated because is a good value on average for determine if a drying pretreatment is necessary, although it is true that some technologies can process waste with more moisture content. FIGURE 2 The figure is not enough clear. The relations between different stages of strategy are not clear. Furthermore, it doesn't reflect the real content of the paper, that is totally lacking about discuions of the obtainable final products or pyrolysis process. Are the authors sure that the proposed strategy can be suitable also for the pyrolysis process and its main drawbacks? Response: Figure 2 has been modified according to the reviewer's comments. FIGURE 7 The figure needs to be restructured. For each box can be inserted the energy content (LHV) of the input stream and its amount, in order to know the energy efficiency of each step. Furthermore, the indication of the process parameters utilised for each step can be shown. Response: Figure 7 has been modified according to the reviewer's comments.

STRATEGY FOR THE DESIGN OF WASTE TO ENERGY PROCESSES BASED ON PHYSICOCHEMICAL CHARACTERISATION

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Abstract
Energy recovery from wastes is needed for cost-effective and sustainable management. For a given waste, the definition of suitable thermochemical conversion process schemes relies on devising a strategy based on several variables among which feedstock characterization is crucial. Depending on the properties of the fuel, the available waste resource may not be suitable for a specific application, for technical and sometimes for environmental reasons [1]. Within this framework, agro-industrial wastes (grape stem, beer bagasse and orange juice residues) were characterized and the results are used to design a strategy for their effective integration in waste-to-energy processes. Energy content, proximate and ultimate analysis, composition, ash fusibility and thermal behaviour were determined. For the physicochemical analysis UNE standard methods were used. Characterization results showed that the three wastes have good quality for thermochemical conversion with energy contents between 19 MJ/kg (beer bagasse) and 16 MJ/kg (orange juice residue) and ash contents below 10% in all cases. However, some drawbacks were found: high moisture (76%), nitrogen (3.5%) and sulphur (0.2%) content for beer bagasse; elevated nitrogen (1.1%) and sulphur (0.15%) concentration for grape stem and nitrogen (1%) content for orange juice residue. All this information has been used to design a smart strategy for selecting a sustainable and environmental friendly waste to energy processes as part of a circular economy approach.

Statement of novelty
This article provides a smart strategy for the selection of the most suitable waste to energy process for waste managers and energy producers within the global concept of circular economy.

Keywords
Waste to energy strategy, fuel characterisation, wastes valorisation, agro industrial waste.

Introduction
Energy recovery from wastes is needed for cost-effective and sustainable management, while it contributes to renewable energy generation or production of high-value chemicals [2]. Waste processing into energy is not only a method of obtaining a valuable product but also a simultaneous way to eliminate a waste storage problem. Energy can be obtained from wastes through several techniques which can be classified in thermochemical, chemical o biochemical processes being thermochemical processes the most commonly used trough the world [3]. Among thermochemical processes, combustion, gasification and pyrolysis are the three main technologies available being combustion the most widely used. Main differences between them refer to the amount of oxygen available during the conversion process: (1) Combustion is the complete oxidation of the residue throughout a series of chemical reactions which transformed the fuel in carbon dioxide and water. Minor components of the fuel (sulphur and nitrogen) can react with air oxygen and form SO_2 and NO_x producing environmental problems if there is not an adequate cleaning system; (2) Gasification is the thermal degradation of a fuel in the presence of an oxidant agent (incomplete combustion). In gasification processes a gas called syngas is obtained which principal constituents are CO, H_2, CH_4, CO_2 and N_2. Gasification is a more versatile technology than combustion due to the fact that with the syngas obtained it is possible to produce liquid fuels, chemicals, electricity or/and heat; (3) Pyrolysis is the thermal degradation of a fuel in absence of an oxidant agent. Three different product phases are obtained with this process, a solid called charcoal, liquids (bio-oil) and gases. Depending on the process conditions the maximization of one of the phases is achieved, normally charcoal or bio-oil.
In order to help in the selection of the most adequate technology for a specific type of waste the aim of this work is to design a smart strategy to be used in sustainable and environmental friendly waste to energy processes as part of a circular economy approach. This selection process depends on many variables that in this work are classified in two types: intrinsic (composition and energetic value of the waste feedstock) and specific (availability of feedstock in sufficient quantity, final product of interest, emissions requirements, energetic and economic balance). Most of the times waste producer differ from energy producer (including heat, electricity, H_2, added value chemical, etc.) so a common strategy that can help to put those two sides together to optimize a waste to energy process is required. In this line for example synergy and symbiosis can be found between wastes from different sources which are one of the goals of the RETOPROSOT-CM project [4]. In this work a smart strategy has been designed where both types of variables (intrinsic and specific) are taken into account in order

to help waste managers and energy producers in the selection of the most adequate approach to optimized waste to energy processes. The strategy includes the definition of the conversion process steps but also blending with cleaner feedstocks so that acceptable solid recovered fuels can be produced. The proposed strategy has been validated using three different wastes and results are presented in this paper.

As far as the authors knowledge, the strategies for waste to energy reflected in the literature consider only in the technology used (basically incineration) with the economical and environmental aspects [5-7], but no a simple strategy that considers all aspects of the process, from the generation of the waste to the application of the final product, has not been found. For this reason, a smart strategy that covers the entire process (waste generated, technology used, technical and environmental requirement and economic aspects) has been designed. To simplify the use of the strategy designed, it has been divided into two parts. The first one considers the characteristics of waste generated as well as the valorisation technology and the second part integrates technological, environmental and economical aspects.

This paper is focus in the first part of the strategy which is related to the physicochemical characteristics of the proposed waste as feedstock the to be used in waste to energy processes.

When considering thermal valorisation of wastes, feedstock properties is the first issue that must be taken into account due to their direct influence on its thermal behaviour. Therefore, fuel characteristics are the first parameters to be considered when a thermochemical technology is selected to produce energy. Depending on fuel properties a specific process or feedstock can be excluded as a suitable option for technical or environmental reasons [1,3]. Regarding the chemical properties of fuels for thermochemical conversion processes the most relevant ones are proximate analysis, ultimate analysis and heating value [3] although other considerations must be taken into account such as inorganic elements composition which are related to corrosion and ash agglomeration.

Proximate analysis is one of the most common methods used for fuel characterization and it is very important when considering thermochemical conversion [3,8]. With proximate analysis the determination of the moisture, ash, volatile matter and fixed carbon was obtained and these parameters are extremely important to predict the thermal behaviour and to design the conversion plant. High moisture content not only decreases the combustion yield, but also increments the cost of transportation. Although in combustion processes fuels with up to 50% of moisture can be processed [9], in gasification fuels with more than 30% of moisture are difficult to process and the preferable moisture content is between 10 – 15% [10]. Ash has great influence in the cost of the processes due to transport and management and also in the corrosion and slag formation [8]. Although there are commercial reactors that can cope with higher ash contents, in general, it is recommended to keep ash content below 6% to reduce the possibility of slag formation. As the ratio volatile matter/fixed carbon is related to fuel's reactivity, high values of this ratio are desirable.

Together with the information provided by the proximate analysis, other parameters such as ultimate analysis [11,12] or calorific value [13-16] can be obtained using regression and correlation methods.

In ultimate analysis the carbon, nitrogen, hydrogen, sulphur, chlorine and oxygen are determined. With this information the calorific value, product´s composition and the environmental impact can be obtained [17]. Carbon and hydrogen are the principal responsible for the calorific value of the fuel while nitrogen, sulphur and chlorine can react to form NH_3, SO_x, H_2S, HCl and/or NO_x which not only may produce corrosion in the equipment but also create environmental problems due to emissions [8,10].

The identification and quantification of inorganic elements in the waste is important in order to predict corrosion problems, ash melting and particulate emissions. Concentrations of major elements such as Ca, K, Mg, P… are relevant regarding to ash melting, slag formation and corrosion while minor elements such as Hg, Cd, and Zn have especial importance in particulate emissions [17,18]. Moreover, inorganic elements can have a catalytic or inhibiting effect in the processes reactions.

Regarding chlorine corrosion problems, there are three main mechanisms that can occur: gas phase corrosion due to HCl or Cl_2, formation of alkali sulphate and/or alkali chloride melting and active oxidation due to sulfidation of alkali metal or heavy metal chlorides [19,20]. Among these mechanisms the most critical one is the active oxidation in high temperature corrosion [21]. However, chlorine corrosion problems can be predicted somehow by applying different indices. The sulphur to chlorine molar ratio (2S/Cl) is an example. If this ratio is high, greater than 8, a protective sulphate layer could be form in tubes minimizing corrosion.

Ash sintering is one of the main sources of failure in conversion plants. Ash agglomeration inhibits fluidisation, reduces heat exchange and might produce the mechanical failure of installations [22]. With the aim of predicting ash sintering, theoretical methods as well as empirical methods have been developed. Among empirical methods, fusibility temperatures determination is used. It consists of heating up an ash pellet and monitoring shape changes. Although laboratory ash is different from real ash produced in a thermochemical industrial process, it can be generally agreed that when the higher the fusion temperatures the lower the probability of slagging [23]. Based on fusibility temperatures an index was described by Gary and Moore [24]. The index is defined as showed in equation 1 and ash behaviour is classified as medium slagging propensity when Fs lies between 1230 – 1342 ºC, high when Fs is 1052 – 1232 ºC and severe if Fs is below 1052 ºC [24].

(1)

$$F_s = \frac{4 \cdot DT + HT}{5}$$

Where DT is the deformation temperature and HT is the hemisphere temperature.

Ash sintering is also related with the amount of alkaline content in the fuel regarding the formation of eutectics with low melting points [25]. In general, the presence of basic oxide compounds lower the melting temperature and that of acidic ones tends to increase it [24]. Taken this into account, several indices have been developed. Some of them are collected below [24,22,26-28].

(2)

$$R = \frac{CaO + MgO}{K_2O + Na_2O}$$

(3)

$$R_b = Fe_2O_3 + CaO + MgO + K_2O + Na_2O$$

(4)

$$R_{b/a} = \frac{Fe_2O_3 + CaO + MgO + K_2O + Na_2O}{Al_2O_3 + SiO_2 + TiO_2}$$

(5)

$$R_{\frac{b}{a}(+P)} = \frac{Fe_2O_3 + CaO + MgO + K_2O + Na_2O + P_2O_5}{Al_2O_3 + SiO_2 + TiO_2}$$

(6)

$$R_{\frac{b}{a}x\,Na} = \frac{Fe_2O_3 + CaO + MgO + K_2O + Na_2O}{Al_2O_3 + SiO_2 + TiO_2} \cdot Na_2O$$

(7)

$$S_R = \frac{SiO_2}{SiO_2 + Fe_2O_3 + CaO + MgO} \cdot 100$$

(8)

$$F_u = \frac{Fe_2O_3 + CaO + MgO + K_2O + Na_2O}{Al_2O_3 + SiO_2 + TiO_2} \cdot (Na_2O + K_2O)$$

(9)

$$R = \frac{Si}{Ca + Mg}$$

(10)

$$R = \frac{Si + P + K}{Ca + Mg}$$

Besides physicochemical properties there are also many other factors that may have a great influence in the conversion process such as operational conditions (type of reactor or temperature). In the case of experimental equipment it depends on each particular case and operational conditions must be optimised for each fuel so general rules are difficult to fix.

2. Methodology

2.1. Strategy designed

To define the most suitable waste to energy thermochemical conversion process applicable to a specific waste a smart strategy has been designed. As briefly described in the introduction section of this paper, besides physicochemical properties of the fuel, which are crucial for thermochemical valorisation design, other considerations must be taken into account for a complete evaluation of the technology such as process conditions, energy balance and economical profitability. Therefore, the proposed strategy has two parts. In the first part, only physicochemical properties are considered to evaluate if a targeted waste is suitable for its thermochemical valorisation and in the second part energy balance, environmental aspects and costs are considered to help in the decision making process.

This paper is focus in the first part of the strategy which is going to be describe in detail below. As is mentioned above in the first part of the strategy only the physicochemical properties of the selected fuel have been considered. Part I of the proposed strategy allows identifying the adequacy of the proposed waste feedstock to be used in waste to energy processes. Three waste to energy processes are kept in mind: combustion, gasification and pyrolysis. When applying the strategy for a specific waste feedstock several considerations must be taken into account such as the amount and shape of waste generated, the heating value, the composition, etc. Many of these properties are related to each other and, depending on the selected thermochemical conversion process, the requirements of these properties will be slightly different. Therefore, the first step is to accomplish the complete characterization of the waste and then evaluate all the properties together considering each time one thermochemical conversion process.

Although operation parameters of the process (type of reactor, temperature, pressure, etc) are important when evaluating the thermochemical process, the first part of the strategy aims to limit the type of process, the pre-treatment requirements for this process and cleaning necessities based on the physicochemical properties.

For pyrolysis process, the main properties considered are the moisture content of the feedstock which should be less than 15% to minimized the production of water and the particle size because the need for rapid heat transfer through the particle. Usually, pyrolysis technologies can process small particles to a maximum of 2 mm [29]. Other important parameter for pyrolysis is the presence of alkali metals (potassium, sodium and calcium) in high concentration due to their catalytic properties. These act by causing secondary cracking of vapours and reducing liquid yield and liquid quality [30]. And for combustion and gasification, moisture content, calorific value and ash composition are the main criteria for suitability for thermochemical processing. As an example, some of the parameters considering in the strategy are reflected in tables 1, 2 and 3.

Table 1. Main fuel specifications depending on gasification reactor type.

Table 2. Typical fuel properties for wood combustion techniques. Adapted from [31].

Table 3. Contaminant values for combustion process. Adapted from [32].

Figure 1 depicts the first part of the designed strategy. In this flow-decision chart blue lines show fulfilment of requisites or conditions where red lines stand for failed criteria.

Figure 1. Simplified flow diagram of Strategy. Part I.

At the end of this part, a first approach of the best waste to energy process is obtained including the pre-treatment requirements and some cleaning and/or upgrading steps. This information is used as an input in the second stage (Part II of the strategy) where the waste to energy technology is selected as well as the product of interest. Although this part of the strategy is out of the scope of the present paper and will be presented in detailed in a future work, in figure 2 a simplified diagram of part II strategy is presented. In summary in this second part of the strategy energy and economic aspect are included to determine if the selected product is generated in a profitable way considering different operational parameters. Cleaning requirements and by-products disposal are also considered according to the currently available technologies.

Figure 2. Diagram of the strategy. Part II.

2.2. Waste feedstocks

Three agro-industrial wastes (grape stem, beer bagasse and orange juice residue) were obtained from Spanish companies. Grape stem consists of branches of the brunch of the grapes which are obtained after the destemming of the grapes in red wines and after the pressing in white wines. Beer bagasse is formed by the mixture of the husk that covers the original barley malt grain and part of the pericarp and seed coat layers that are obtained as residuals solid mater after the wort extraction step [33]. Finally, orange juice waste consists of orange skin and pulp obtained after juice extraction.

2.3. Physicochemical characterization

A complete physicochemical characterization of the three proposed samples was carried out in the Biomass Laboratory of CEDER-CIEMAT following the lasted European standards for biomass feedstock that are summarized in table 4.

Table 4. Standards methods used for physicochemical characterization.

First of all, samples were prepared for the analysis by means of homogenization, grinding and drying to ensure good quality samples. Measurements were done in duplicate and mean results were expressed in dry bases except for moisture content.

Proximate analysis

Proximate analysis is one of the most common methods for fuel characterization and it is very important when considering thermochemical conversion [3,8]. With proximate analysis the determination of the moisture, ash, volatile matter and fixed carbon was obtained and these parameters are extremely important to predict the thermal behaviour and to design the conversion plant. Moisture, ash and volatile content were obtained following international standard while fixed carbon was calculated by difference. The volatile matter was obtained following the standard UNE-EN ISO 18123:2016 which consisted of heating up the sample to 900 ºC in absence of air. Moisture was determined by heating the sample at 105 ºC until constant weight as indicated in the UNE-EN ISO 18134-2:2016 standard. Ash content was determined by burning the sample at 550 ºC using different fixed heating rates to minimize the volatilisation of inorganic elements in a conventional laboratory furnace according to UNE-EN ISO 18122:2016. And fixed carbon was calculated by difference subtracting from 100% the sum of the volatile matter and ash content.

Ultimate analysis

According to UNE-EN ISO 16948:2015, the analysis of carbon, nitrogen and hydrogen was carried out in a TruSpec (Leco) elemental analyser where the sample is combusted at 950 ºC in oxygen atmosphere and the resulted gases where analysed with infrared detectors for carbon and hydrogen determination and with a thermal conductivity detector (TCD) for nitrogen determination after reduction and cleaning steps.

The determination of chlorine and sulphur was done by ionic chromatography after the recuperation of the liquids precedent of a bomb combustion following the UNE EN ISO16994:2017 standard.

Finally, oxygen content was calculated by subtracting from 100% the sum of carbon, hydrogen, nitrogen, sulphur, chlorine and ash contents.

Determination of inorganic elements

The determination of inorganic elements is important not only to prevent operational problems such as ash melting or slugging but also to predict particulate emissions and environmental problems.

The determination of major inorganic elements such as aluminium, calcium, iron, magnesium, phosphorus, potassium, silicon, sodium or titanium was done according to the following procedure: first, samples of ashes were obtained using the aforementioned procedure, then they were digested in a microwave oven according to UNE-EN ISO: 16967:2015 standard and finally they were analysed with inductively coupled plasma atomic emission spectrometry (ICP-AES) (THERMO JARRELL ASH simultaneous spectrometer). Minor elements were obtained in a similar way following UNE-EN ISO 16968:2015 standard.

Determination of calorific value

High heating value at constant volume in dry basis was obtained following the UNE-EN ISO 14918:2011 standard using a LECO AC-300 calorimeter. Low heating value is calculated according to the same standard.

Ash fusibility

Fusibility temperatures that characterise ash melting behaviour were determined according to CEN/TS 15404. This method consists of two steps. (1) moulding a cylindrical pellet (3 mm height and diameter) with ashes powered until they pass a 0.25 mm sieve and (2) heating up the pellet in a furnace with oxidising atmosphere (air) from room temperature to 1400 ºC using a fixed heating rate. With an optical heating microscope (LEICA), four temperatures were recordered based on changes in the shape of the pellet during the heating process. The characteristic temperatures measured were: shrinking temperature (ST) defined as the temperature at which the sample area becomes less than 95% of the original area at 550 ºC, deformation temperature (DT) defined as the temperature at which the first signs of rounding of the edges due to melting of the test piece occurs, hemisphere temperature (HT) defined as the temperature at which the test pellet forms approximately a hemisphere and flow temperature defined as the temperature at which the ash is spread out over the supporting title in a layer.

2.4. Thermal analysis

Thermogravimetric analysis (TGA) was done to complete the characterization analysis. Measurements were realized in the Combustion Laboratory at CIEMAT using a Mettler TGA/SDTA 851e (Mettler Toledo Corporation, Switzerland) thermal gravimetric analyser. For the analysis, around 40 mg of dried sample grinded between 0.1 and 0.2 mm were used. TGA was done at three different heating rates (5, 10 and 20 ºC/min) form 30 ºC to 900 ºC in a nitrogen atmosphere (50 mL/min).

3. Results

3.1. Wastes

The amount and form in which the residues are available is an important factor when their thermochemical valorisation is studied. In figure 3, the different visual aspect (shape, particulate size, etc.) of the residues used in this work for the validation of the proposed strategy can be observed.

Figure 3. Physical aspect of residues.

In the specific cases used in this work beer bagasse is obtained as a homogeneous wet residue in a limited quantity due to the fact that the producer is a local craft brewery and a drying step is necessary prior to its thermochemical valorisation. In the case of the grape stem, this residue is available in huge quantities but it is produced in a seasonal basis and in a form that precise a grinding and homogenization steps. Finally, the orange juice residue is produced in a seasonal basis and in an almost homogeneity form so in principle no grinding neither homogenisation steps will be mandatory.

3.2. Physicochemical characterisation

Table 5 summarizes the main parameters obtained during the physicochemical characterisation of the three wastes considered in this work. Proximate analysis of all fuels showed medium volatile content (73 – 79%) so a rapid conversion of solid products into gaseous ones could be expected. Ash content is relatively low in all cases (3 – 6%), therefore no problems due to ash sintering are expected but this fact will be studied deeply later. Regarding moisture contents, only in the case of beer bagasse this value is very high so for its thermochemical valorisation a dry pre-treatment will be necessary.

All residues presented good calorific value in dry bases, the lowest value was obtained for orange juice residue (HHV: 17 MJ/kg) which is similar to other agroindustrial residues such as mill bagasse [34].

Ultimate analysis showed high levels of sulphur for beer bagasse (0.24%) and for grape stem (0.15%) and quite high nitrogen content for all residues especially for beer bagasse (3.5%). In consequence, some cleaning systems, especially in the beer bagasse case, must be considered when designing the thermochemical process to fit environmental regulations and/or quality specifications of downstream processes.

Corrosion in conversion plants due to the presence of chlorine can be predicted with the sulphur to chlorine molar ratio using equation 11. When this ratio is higher than 4, only minor corrosion could be expected due to the form of a protective sulphate layer on surface tubes [21,19].

$$I = 2S/Cl \qquad (11)$$

High values for sulphur to chlorine molar ratio have been found for beer bagasse (I = 13) and for grape stem (I = 17) therefore no corrosion problems could be expected. Nevertheless, in the case of orange juice residue this index is quiet low (I = 2) so special care must to be taken into account in the plant design.

Major inorganic elements were analysed in ashes and expressed as oxides in figure 4. The most abundant constituents were phosphorous, siliceous, magnesium, calcium and potassium for beer bagasse, calcium, potassium and silicon for grape stem and calcium and potassium for orange juice residues. Many indexes have been described based on the relation between alkaline earth and alkaline oxides to predict sintering tendency as it has been mentioned above. One of them is reflected in equation 12. It can be said that biomass with R values higher than 2 should not present risk of sintering [22,26].

$$R = (CaO + MgO) / (K_2O + Na_2O) \qquad (12)$$

For the residues studied in this work, index R showed a value of 3 for beer bagasse and orange juice residue and 5 for grape stem. Therefore the expected sintering tendency is low for all of them. This fact is corroborated with the fusibility temperatures which are high for all the residues as reflected in table 5.

Table 5. Physicochemical characterisation.

Figure 4. Oxides in ashes

3.3. Thermal characterization

As an example, in figure 5, DTG curves at 20 ºC/min are presented. DTG curves show that although all residues have similar elemental compositions, they have different degradation patrons. All residues exhibit the typical degradation patron of lignocellulosic materials which are represented by the degradation of its principal constituents (cellulose, hemicellulose and lignin), but some differences can be noticed.

Figure 5. DTG curves at 20 ºC/min

In the first step, common for all samples, the loss of the residual moisture can be appreciated up to around 120 ºC. Between 200 ºC and 320 ºC decomposition of cellulosic materials occurs and finally the degradation of the lignin takes place at temperatures around 350 ºC. The most differentiated thermal behaviour was presented by orange juice residue with two important mass losses between 120 – 230 ºC (20%) due to the loss of volatile compounds and between 285 – 368 ºC (23%). In the case of grape stem, the major lost takes place between 290 – 380 ºC (31%) probably associated with the decomposition of lignin which is the principal constituent of grape stem [35]. Regarding to beer bagasse, the major loss takes place between 240 – 320 ºC (27%). At lower temperature, losses are very low due to the fact that the beer bagasse was obtained after a thermal process at low temperature so more thermolabile compounds were lost. At high temperature, two losses were observe around 350 ºC (19%) and 380 ºC (16%). Therefore the beer bagasse residue needs the highest temperature to reach the complete degradation.

3.4. Validation of the proposed strategy for thermochemical process selection

The proposed strategy has been validated by using the three waste samples considered in this work. Figure 6 shows the results of applying part I of the strategy to the three wastes studied. According to the physicochemical characterization, the three wastes exhibited good potential for thermal valorisation following a waste to energy approach although a different pathway is required in each case. According to the strategy proposed orange juice waste could be used following the most straightforward pathway while grape stem would require a pre-treatment and beer bagasse a drying process. Besides that all wastes showed significate sulphur and nitrogen contents which must be taken into account during the application of the second part of the strategy.

Figure 6. Simplified flow chart for the three wastes studied.

After the confirmation of the suitability of the proposed waste for its thermochemical valorisation using Part I of the strategy, Part II was used to guide in the selection of the most suitable technology and its technoeconomical viability. For validating part II of the strategy, considerations were taken into account such as the amount of residue generated, the pre-treatment necessities and the cleaning or conditioning requisites for the final product desired. Beer bagasse produced in a local brewery in Spain has been selected as example for validating the strategy. The local brewery has an annual beer production of 5040 L and generates 100.8 T/y of beer bagasse (BSG) with 76% of moisture. To generate the heat necessary for the brewering process this company uses a diesel boiler to generate steam. Therefore, in this case targeted final product is heat to be used in the same process, so following the proposed strategy two different technologies could be used: combustion and/or gasification.

Application of Part I of the strategy confirmed that beer bagasse can be a suitable waste for being used in a waste to energy process but additional requisites of pre-treatment and gas cleaning (high contents of sulphur and nitrogen) would be required. Then, following part two of the strategy, emissions requirements were checked and cleaning requirement identified for both approaches (combustion and gasification). Basic energy balance including all energetic penalties due to pre-treatment and gas cleaning steps were performed and summarized in figure 7.

Figure 7. Scheme with energy balance.

Gasification of beer bagasse for a circular economy application has been studied previously in our group by Ortiz et al. [36] for the same type of industry. In this work two final products were considered: heat and electricity. With the aim of validate the strategy, the simplest case (heat production) was considered. In their study the gasification technology selected was an atmospheric fluidized bed gasifier which used air as gasification agent and the beer bagasse pre-treatment was carried out by a drying step with a hybrid solarbiomass greenhouse following by a densification step by pelletization process. According to this work, the energy necessary for the pre-treatment process is 11.4 MWh per year while the energy produced by the gasification technology is 69.7 MWh/y.

Regarding to the combustion process, the work carried out by Charles W. Morrow [37] was considered. In this study he founds that with a feeding rate of 50 lb/h of dry bagasse 90 kWh of energy can be produced to run a boiler. Transferring this data to our particular case where 21.8 T/y of dry bagasse is produced after pre-treatment process, the energy that can be provided to a boiler is 86.5 MWh/y. The energy consumption of the combustion process necessary for BSG feeding and pumps movement is consider equal to the energy consumption for gasification process. Although in the work by Harles W. Morrow the gas cleaning is not considered, the beer bagasse composition suggest the necessity of a cleaning step to comply with emissions requirements. For this work the simplest cleaning system was considered: a cyclone and a guard bed with active carbon so only energy penalties due to heat losses were estimated. In a good isolated system, minimal heat losses can be produced so only 0.1 MWh/y was considered.

Comparing processes, gasification and combustion, the energy that can be obtained in the particular case studied, is greater with the combustion processes. Although to make a final decision economical consideration must be taken into account even though they are out of the scope of this work.

Conclusions

In this work a two-part smart strategy for the analysis of the suitability of different wastes for their thermochemical valorisation to energy following a waste to energy technology has been proposed. Part I allows deciding if a specific waste is suitable for being used in a waste to energy process based on its physicochemical properties. Part II summarises the most relevant parameters that should be taken into account within a circular economy approach where energy balance, environmental requisites and economical balance are included. For validating the proposed strategy three wastes have been studied in this work: beer bagasse, grape stem and orange juice residue. Main properties of those wastes were obtained and used in the strategy. According to the physicochemical characterization, the three wastes exhibit good potential for thermal valorisation and specific requisites had to be taken into account for selecting the most adequate waste to energy technology, such as the moisture content of beer bagasse which might require a previous drying step or its sulphur and nitrogen content which dictate strong gas cleaning requirements, the grape stem form which require a gridding pre-treatment and gas cleaning conditioning due to its nitrogen and sulphur content and in the case of orange juice residue, only cleaning steps due to its nitrogen content will be necessary.

A case study using beer bagasse produced in a local brewery in Spain was used in this work as waste stream for validating the strategy. Heat was selected as final product of interest. According to the proposed strategy two waste- to-energy technologies could be used: combustion and gasification. A specific analysis of the energy requirements of the process and the gas cleaning requirements allowed selecting the most adequate solution from a technical point of view. An economical evaluation, not included in this work, would provide the final decision.

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Figure 1

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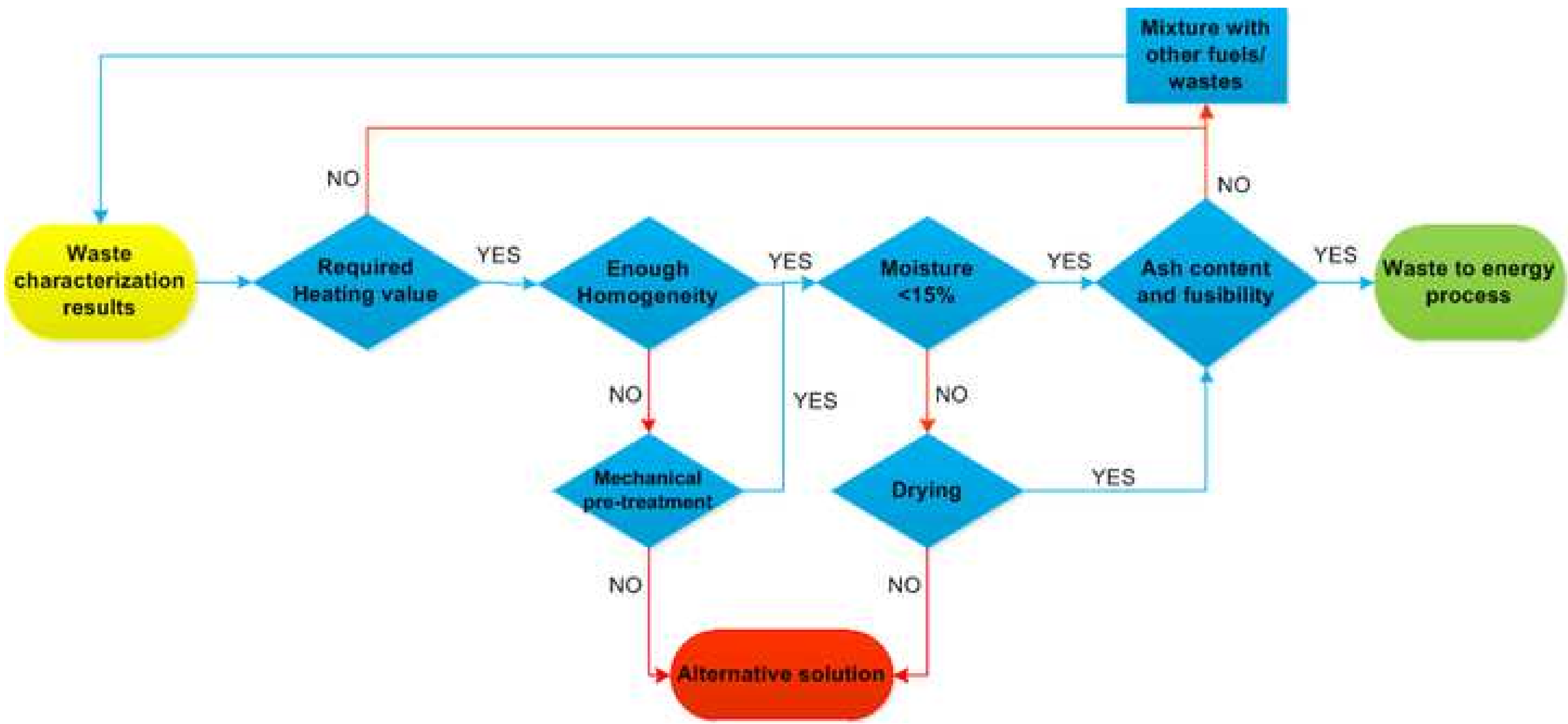
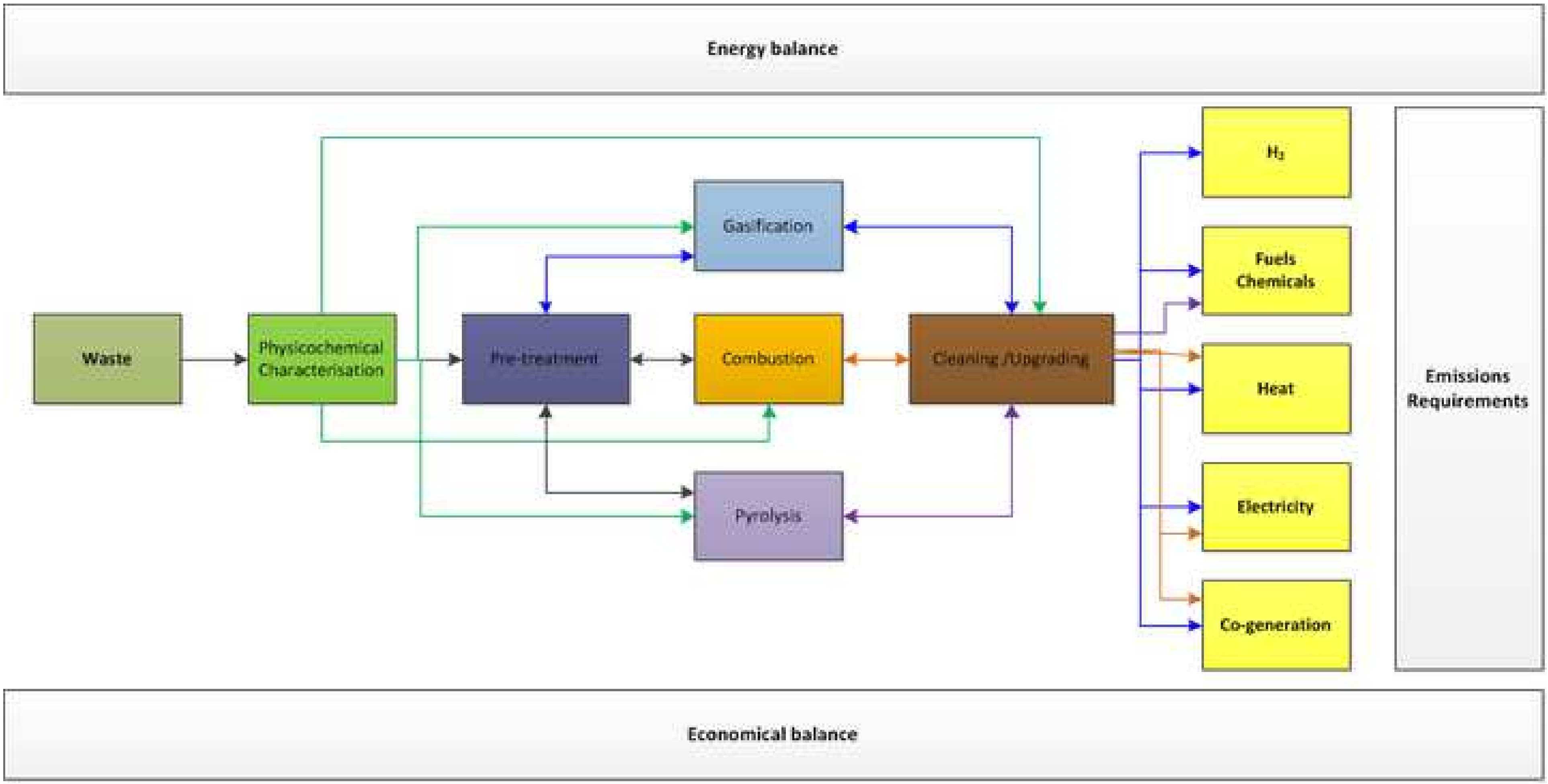


Figure 2



Energy balance
Waste
Physicochemical Characterisation
Pre-treatment
Gasification
Combustion
Pyrolysis
Cleaning /Upgrading
H_2
Fuels Chemicals
Heat
Electricity
Co-generation
Emissions Requirements
Economical balance

Figure 3

Figure 4

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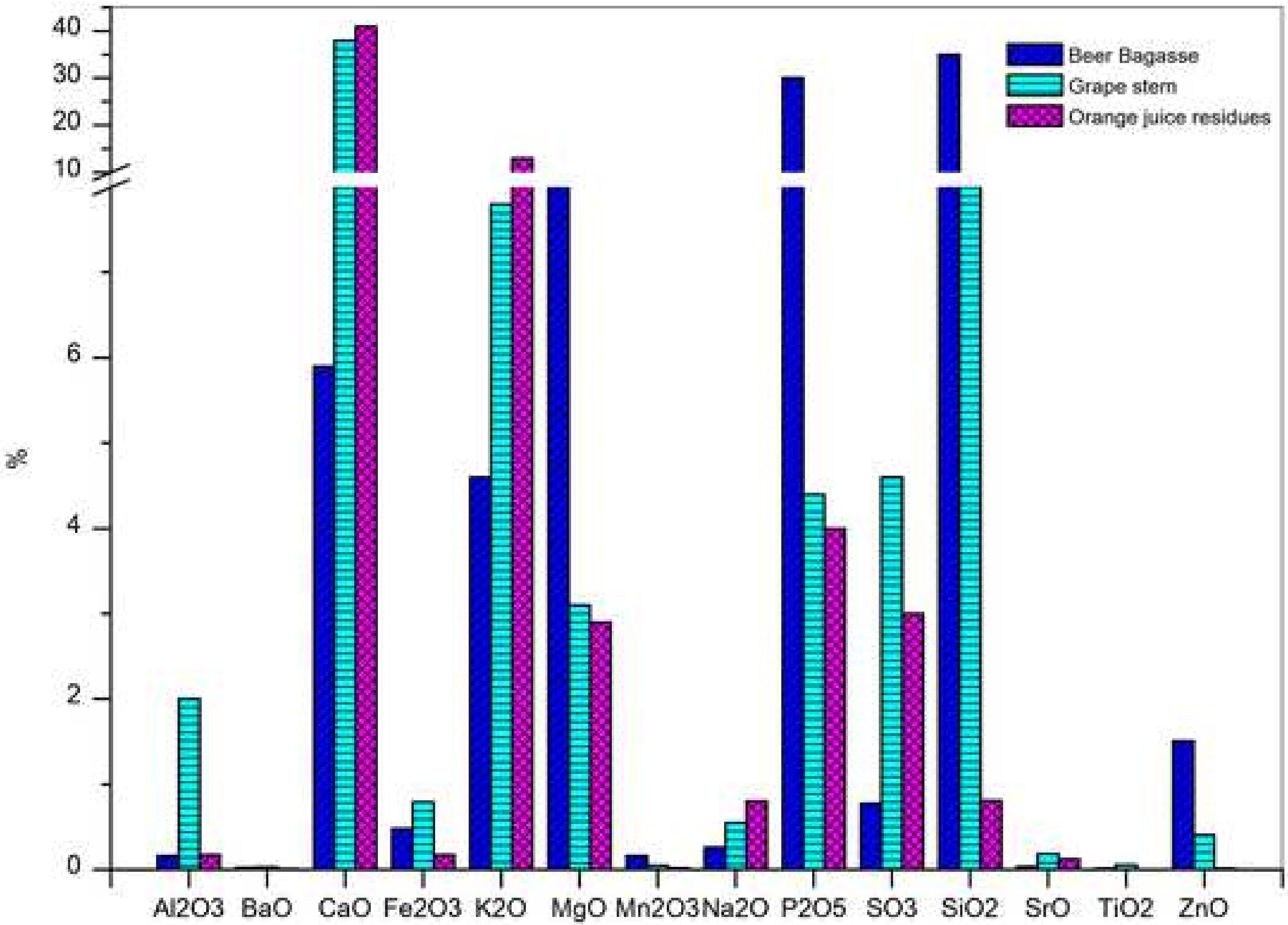

Figure 5

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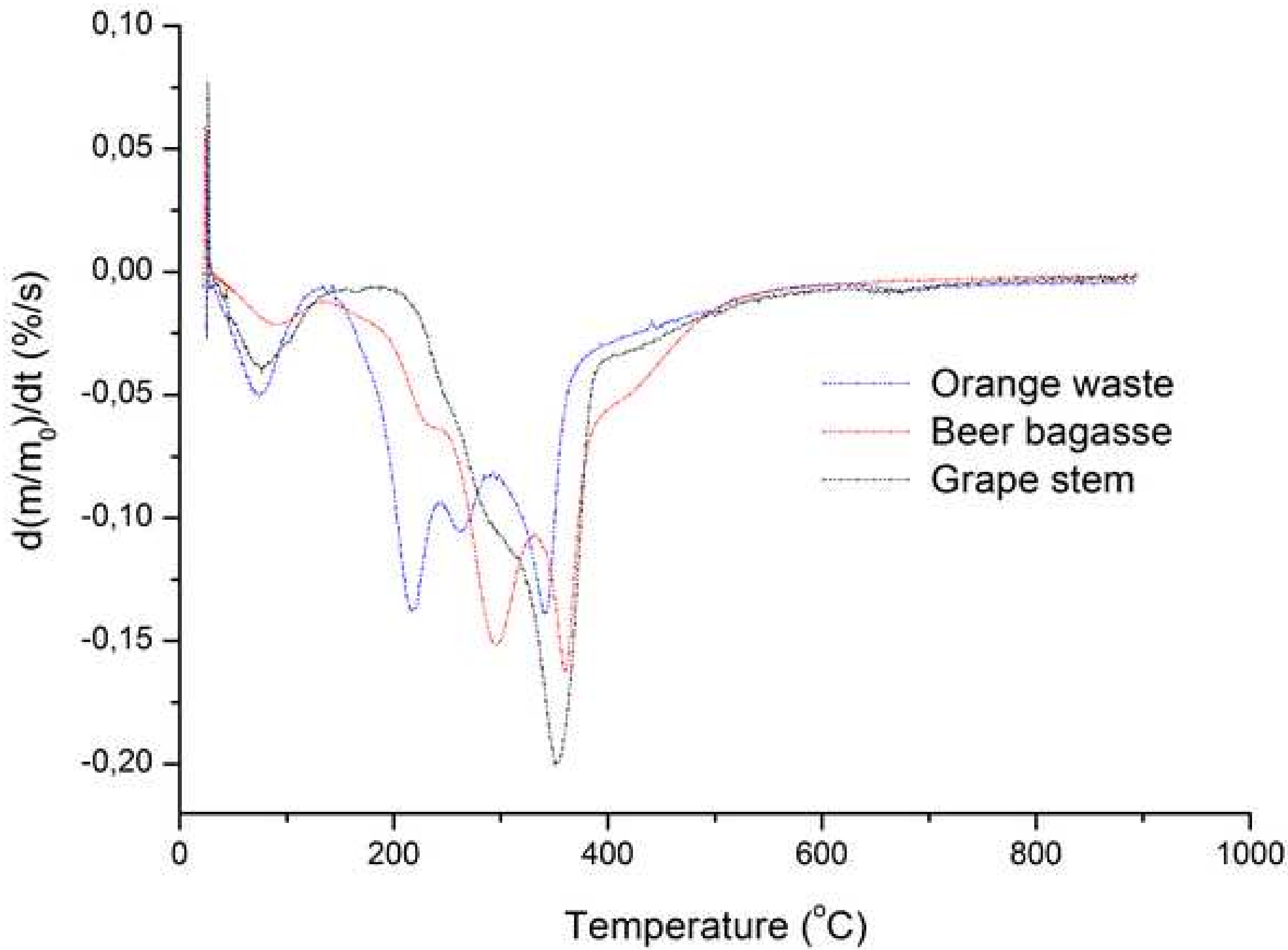

Figure 6

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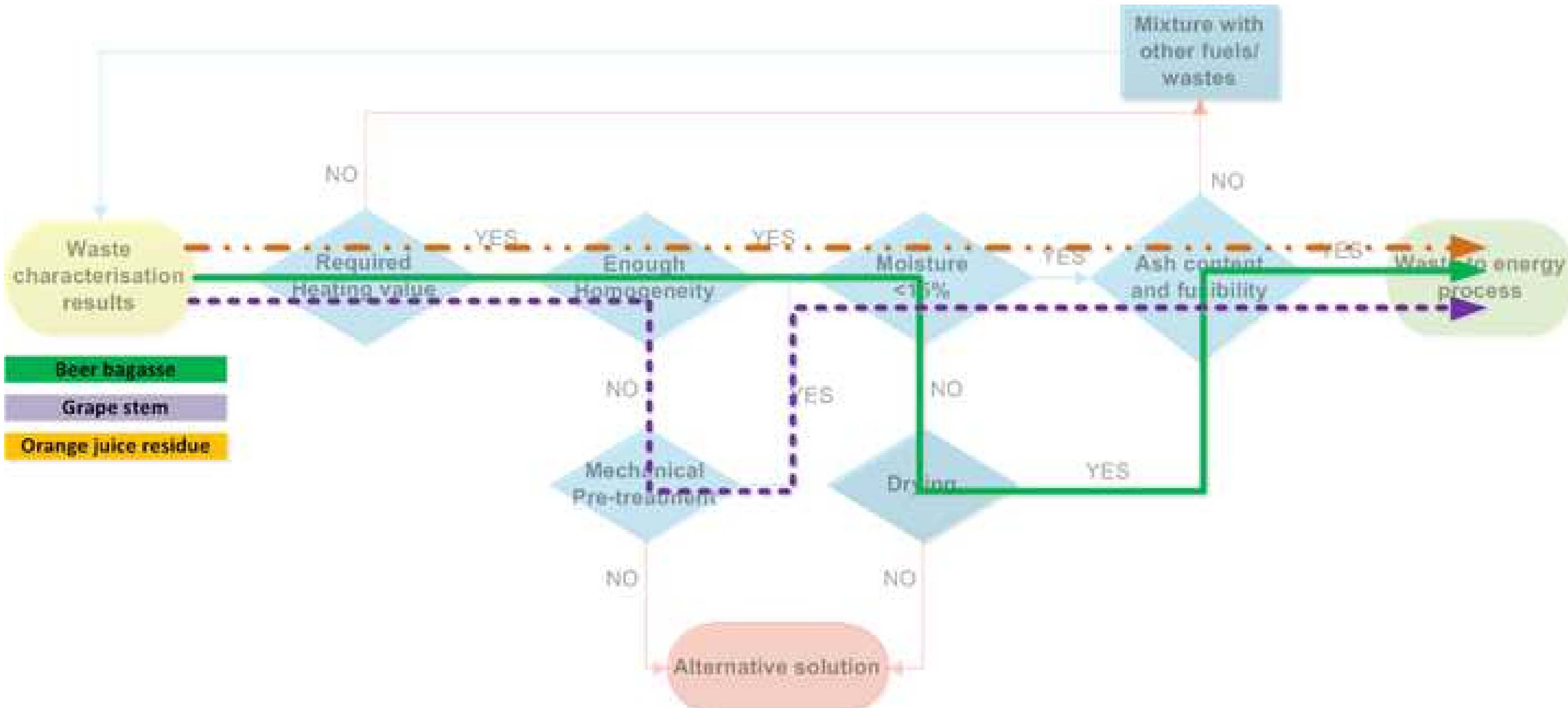

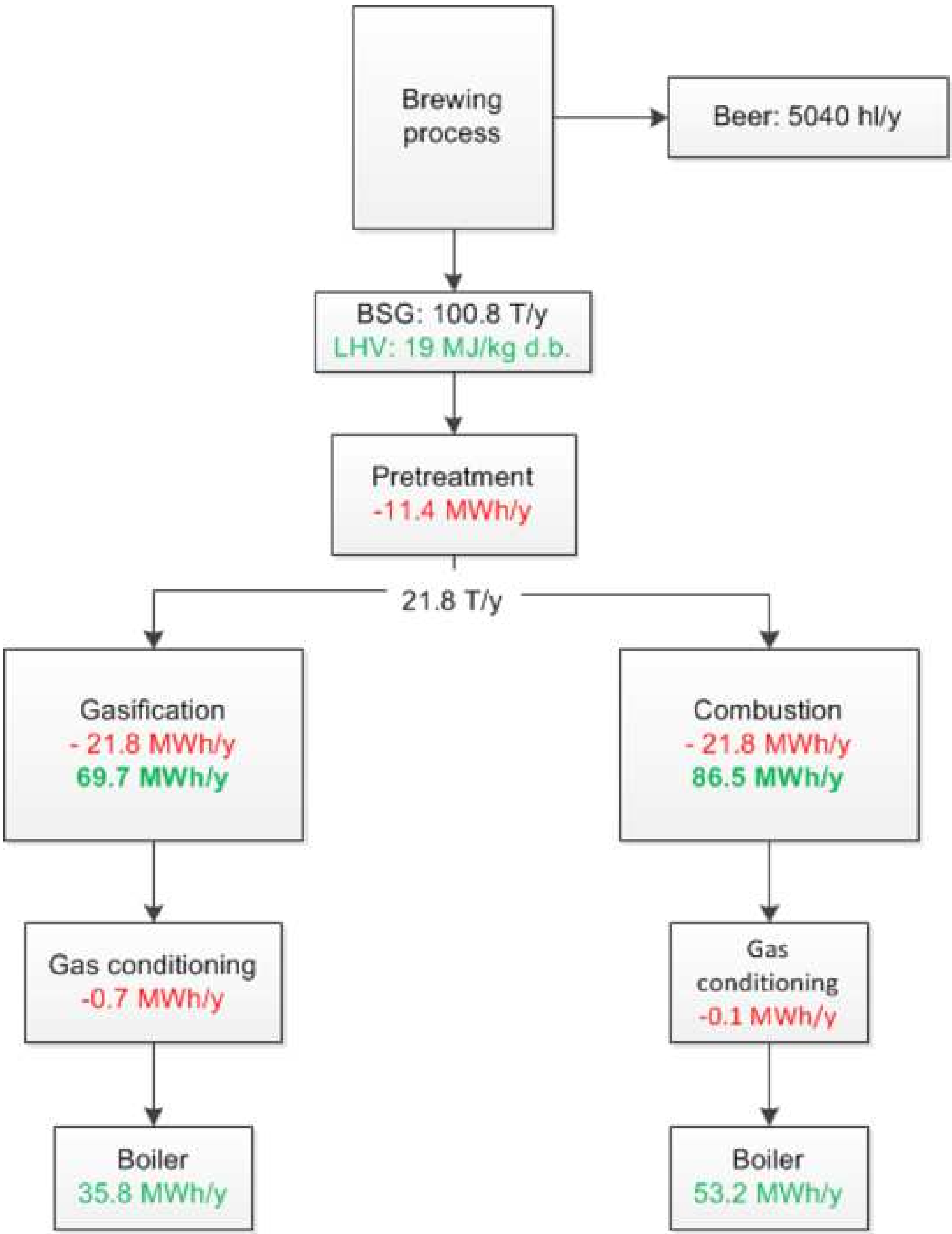
Brewing process
Beer: 5040 hl/y
BSG: 100.8 T/y
LHV: 19 MJ/kg d.b.
Pretreatment
-11.4 MWh/y
21.8 T/y
Gasification
- 21.8 MWh/y
69.7 MWh/y
Combustion
- 21.8 MWh/y
86.5 MWh/y
Gas conditioning
-0.7 MWh/y
Gas conditioning
-0.1 MWh/y
Boiler
35.8 MWh/y
Boiler
53.2 MWh/y

Table 1. Main fuel specifications depending on gasification reactor type.

Type of reactor	Downdraft	Updraft	Fluidizing
Moisture	< 15%	< 50%	< 40%
Size (mm)	< 1	< 1	< 50 – 150 (< 20*)
Ash (d.b.)	< 5%	< 15%	< 20%
Ash fusion temperature	> 1250 ºC	> 1000 ºC	> 1000 ºC

d.b.: dry basis
*circulating

Table 2. Typical fuel properties for wood combustion techniques. Adapted from [31]

Type	Fuels	Ash	Water content
Dual-chamber	Dry residues	< 5%	5 – 35%
Moving grate	Most biomass	< 50%	5 – 60%
Rotating grate (BFB)	d < 10 mm	< 50%	5 – 60%
Rotating grte (CFB)	d < 10 mm	< 50%	5 – 60%
Entrained flow	d < 5 mm	< 5%	< 20%

Table 3. Contaminant values for combustion process. Adapted from [32].

Element	Value (% d.b.)	Associated issue
Cl	> 0.1	Corrosion
S	> 0.1	Corrosion
K	> 7.0	Ash melting / corrosion / deposits
Na	> 2.0	Ash melting / corrosion / deposits

Table 4. Standards methods used for physicochemical characterization.

Parameter	**Standard**
Proximate analysis	
Volatile matter	UNE-EN ISO 18123:2016
Ash	UNE-EN ISO 18122:2016
Moisture	UNE-EN ISO 18134-2:2016
Ultimate analysis	
C, H, N	UNE-EN ISO 16948:2015
S, Cl	UNE-EN ISO 16994:2017
Calorific value	UNE-EN ISO 14918:2011
Inorganic elements	
Major elements	UNE-EN ISO: 16967:2015
Minor elements	UNE-EN ISO 16968:2015
Ash fusibility	CEN/TS 15404:2010

Table 5. Physicochemical characterisation.

		Grape stem	Beer bagasse	Orange juice residues
Proximate analysis				
Moisture	(wt. % a.r.)	16	76	12
Ash	(wt. % d.b.)	6	3	6
Volatile matter	(wt. % d.b.)	73	79	76
Fixed carbon	(wt. % d.b.)	22	18	18
Calorific Value				
High heating value	(MJ/kg d.b.)	19	21	17
Low heating value	(MJ/kg d.b.)	17	19	16
Ultimate analysis				
Carbon	(wt. % d.b.)	49	49	44
Hydrogen	(wt. % d.b.)	6	7	6
Nitrogen	(wt. % d.b.)	1	3.5	1
Sulphur	(wt. % d.b.)	0.15	0.24	0.05
Chlorine	(wt. % d.b.)	0.02	0.04	0.06
Oxygen	(wt. % d.b.)	38	38	43
Fusibility temperatures				
Shrinking temperature	(ºC)	>1450	680	>1450
Deformation temperature	(ºC)	>1450	>1450	>1450
Hemisphere temperature	(ºC)	>1450	>1450	>1450
Flow temperature	(ºC)	>1450	>1450	>1450

Title Page

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Title:

Strategy for the design of waste to energy processes based on physicochemical characterisation

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Abstract

Energy recovery from wastes is needed for cost-effective and sustainable management. For a given waste, the definition of suitable thermochemical conversion process schemes relies on devising a strategy based on several variables among which feedstock characterization is crucial. Depending on the properties of the fuel, the available waste resource may not be suitable for a specific application, for technical and sometimes for environmental reasons [1]. Within this framework, agro-industrial wastes (grape stem, beer bagasse and orange juice residues) were characterized and the results are used to design a strategy for their effective integration in waste-to-energy processes. Energy content, proximate and ultimate analysis, composition, ash fusibility and thermal behaviour were determined. For the physicochemical analysis UNE standard methods were used. Characterization results showed that the three wastes have good quality for thermochemical conversion with energy contents between 19 MJ/kg (beer bagasse) and 16 MJ/kg (orange juice residue) and ash contents below 10 % in all cases. However, some drawbacks were found: high moisture (76%), nitrogen (3.5%) and sulphur (0.2%) content for beer bagasse; elevated nitrogen (1.1%) and sulphur (0.15%) concentration for grape stem and nitrogen (1%) content for orange juice residue. All this information has been used to design a smart strategy for selecting a sustainable and environmental friendly waste to energy processes as part of a circular economy approach.

Keywords

Waste to energy strategy, fuel characterisation, wastes valorisation, agro industrial waste.

Acknowledgments

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Response to the Reviewers' Comments

Journal: Waste and Biomass Valorization
Article Ref: WAVE-D-18-01045
Title: Strategy for the design of waste to energy processes based on physicochemical characterisation

COMMENTS FOR THE AUTHOR:

Reviewer #1: WAVE-D-18-01045
The paper offers a smart strategy for identifying a suitable thermochemical process for a waste, based on feedstock physicochemical characterisation. The topic appears interesting, but the proposed approach appears not holistic and lacking of some crucial issues. The organisation of the paper should be restructured, in order to better show the innovation of the proposed strategy and its application.

INTRODUCTION

- Most part of the introduction (for example the description of each type of the analysis or the equations 1-10), can be moved in other part of the paper or deleted. In the introduction part of a paper, the crucial points of the topic under analysis should be stated, an overview of state of art should be shown, and the structure of the paper together with the indication of its main objectives should be inserted. For example, are there proposed other "smart strategies" in the already published literature? What is the contribution of this paper in the field?

Response:

The introduction part has been modified taken into account the comments of the reviewer.
The main objectives of the work are in the paragraph: "In order to help in the selection of the most adequate technology for a specific type of waste the aim of this work is to design a smart strategy to be used in sustainable and environmental friendly waste to energy processes as part of a circular economy approach."

METHODOLOGY

- This part should begin with a general description of the smart strategy with the indication of its main steps and support information for each of this step (for example the indication of carried analyses or indexes utilised in each step). To this aim, a clear and schematic figure (but at same time rich of information) could be inserted.

Response:

Methodology section has been modified according to the reviewer's comments.

SECTION 2.4

- Line 43. The selection of a product of interest (hydrogen, electricity, biofuels) cannot be related only to the suitability of a feedstock for a thermochemical process, but also to the technical performances of the process itself. For example, the production of a biofuel (but also of a chemical or hydrogen) requires strict requirements in terms of syngas cleaning and conditioning. Specifically, tar content in the syngas obtained from gasification represents the main issue for the syngas utilisation in final devices able to produce electricity or high added products (fuels, chemicals etc.). How the proposed strategy takes into account tar content and its related issues?

Response:

The product of interest is an input in the strategy. Once selected, the strategy evaluate the suitability considering the process used and the cleaning requirements. In the case of tar cleaning the technology considered in the strategy is based in a scrubber but this aspect is not development in the present paper because it will be included in the second part of the strategy.

- Table 2. The table appears not representative of the main fuel specifications. For example, physical properties of the feedstock (e.g. size and form) could be inserted. Furthermore, not only the requirements of a reactor are crucial for the suitability of a feedstock to the gasification process, but also the conditions (authothermal or allothermal), together with the indication of type of the gasifying agent (oxygen? Air? Steam?.

Response:

Table 2 has been modified according to the reviewer’s comments.

- Table 3. Contaminants content is not the only issue to take into account for the combustion process. The type of reactor is crucial also in this case. For example, a fluidized bed combustor shows most stringent requirements than those of a moving grate furnace. Please, also if the table aims to be "only an example", in the reviewer's opinion, it could be better structured (as already indicated also for the table 2) in order to clarify all types of specifications to be taken into account. The same should be done for pyrolysis.

Response:

Table 3 is only an example but more information has been added in other table according to reviewer’s comments.

- Line 21. How the smart strategy takes into account the environmental aspects? The emissions in the air, water and soil by means of a thermochemical process can be not related only to the conversion stage, but they are strictly dependent from the air pollution control systems. Furthermore, a discussion about the fate of solid residues from processes (based on their composition that strictly depend from waste composition) is totally lacking in the paper.

Response:

This paper is focus in the first part of the strategy which is related to the physicochemical properties of the waste so it does not include details of the second part. Authors are totally agree with the comments of the reviewer, but the discussion of this aspect will be included in future work where the second part of the strategy will be described.

FIGURE 1

The figure appears not clear and self-understanding. Why a moisture content below 15% is indicated? Is this value different for different types of processes? Maybe, it could be more correct to refer to "pre-treatment" as "mechanical pre-treatment" in order to distinguish it from drying stage that can be also a pre-treatment.

Response:

Figure 1 has been modified according to the reviewer's comments. Regarding to moisture content, 15% is indicated because is a good value on average for determine if a drying pretreatment is necessary, although it is true that some technologies can process waste with more moisture content.

FIGURE 2

The figure is not enough clear. The relations between different stages of strategy are not clear. Furthermore, it doesn't reflect the real content of the paper, that is totally lacking about discuions of the obtainable final products or pyrolysis process. Are the authors sure that the proposed strategy can be suitable also for the pyrolysis process and its main drawbacks?

Response:

Figure 2 has been modified according to the reviewer's comments.

FIGURE 7

The figure needs to be restructured. For each box can be inserted the energy content (LHV) of the input stream and its amount, in order to know the energy efficiency of each step. Furthermore, the indication of the process parameters utilised for each step can be shown.

Response:

Figure 7 has been modified according to the reviewer's comments.