Towards food circular economy: hydrothermal treatment of mixed vegetable and fruit wastes to obtain fermentable sugars and bioactive compounds

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ABSTRACT

Due to processing activity, fruits and vegetables generate notable amounts of wastes at the processing, retail, and consumption level. Following the European goals for reducing food wastes and achieving a circular economy of resources, theses biowastes should be valorised. In this work, hydrothermal hydrolysis at different conditions (temperatures, times, waste/water ratio, pH values) were tested to treat for first time, biowastes composed of mixed overripe fruits or vegetables to maximize the extraction of fermentable sugars that can be used as substrates in bioprocesses. Experimental data were fitted by a model based on irreversible first-order reactions, and kinetic constants were obtained. When hydrolysis of fruit wastes was carried out at 135°C and pH 5 during 40 min, more than 40 g of reducing sugars per 100 g of waste (dry weight) could be obtained (represents an extraction of 97% of total carbohydrates). Concentrations of inhibitor compounds (HMF, furfural, acetic acid) in the hydrolysates were very low and, as example, a fermentation to obtain bioethanol was successfully carried out with an efficiency above 95%. Additionally, the production by hydrothermal treatment of bioactive compounds was investigated and the best results obtained were 92% DPPH inhibition and 12 mg GAE/g (dry weight) for antioxidant activity and phenolic compounds, respectively. These values are similar or even higher than those reported in literature using specific parts of fruits and vegetables.

Keywords: Antioxidant capacity**;** fermentation; fruit residues, hydrolysis; polyphenols; vegetable wastes.

1. INTRODUCTION

According to Food and Agriculture Organization (FAO 2011), approximately 1.6 billion tonnes of food for human consumption are wasted every year and more than 80% of these wastes corresponds to the edible part of food. The European Union generates around 88 million tonnes of food wastes per year and this amount is expected to increase by more than 40% in the coming years (Esparza et al. 2020). Particularly, in Spain, more than 1300 million kg of food were discarded in households in 2018, 46% of them were fruit and vegetables (MAPA 2019). Only a small fraction of the food wastes generated at this level is reused or recycled, so that the majority of these residues are landfilled or incinerated (Gustavsson et al. 2011; Nanda et al. 2015). In order to mitigate this problem, the United Nations (ONU), with the support of the European Commission (EU 2015), has proposed a roadmap with the goal of reducing food losses and waste by 50% by 2030 (Joensuu et al. 2021). These actions intend to bring the current environmental state closer to the climate neutrality that would have to be achieved before the end of the century, as it was proposed in the Paris Agreement (Karić 2022).

With a shorter half-life than other food products, large amounts of wastes are generated from fruits and vegetables at different levels (processing industry, supermarket, and household) (Seguí and Fito 2018). Specifically, these wastes are disposed off in large quantities at the points of sale, in some cases exceeding 25% of the product purchased. Besides, it has been reported that the generation and accumulation of organic wastes is one of the main challenges faced by food processing plants (Goula and Lazarides 2015; Seguí and Fito 2018). For these reasons, the interest in investigating new alternatives that allow the valorisation of these organic wastes is increasing.

In a context of circular economy, one option to minimise the volume of organic wastes sent to landfill is to employ these residues as raw material in transformation processes with the aim to obtain value-added products. In this sense, the fruit and vegetable sector is very interesting. This kind of residues, generated at both supermarket and industrial level, are quite homogeneous in terms of characteristics and composition, so that a classification stage is not necessary. Furthermore, fruit and vegetable wastes, with a proper treatment, can be used as substrates in fermentative bioprocesses since they are very rich in carbohydrates (Cekmecelioglu and Uncu 2013; Procentese et al. 2017; Quintero et al. 2011). Hence, these carbohydrates could be used to produce different bioproducts, such as bioethanol whose production has experienced a

significant growth during the last decades and provides a potential alternative of valorisation (Esparza et al. 2020; Yang et al. 2016; Ude et al. 2020).

In addition, fruit and vegetable by-products and wastes represent one of the main sources of bioactive compounds such as polyphenols, which exhibit antioxidant and anti-inflammatory properties. These bioactive compounds could have potential applications in cosmetic, pharmaceutical and food industry (Castrica et al. 2019; Kabir et al. 2015; Wijngaard et al. 2009; Odewale et al. 2021). For example, citrus peel and red fruits such as strawberries are considered an important source of flavonoids (Singh et al. 2020; Vázquez-González et al. 2020). Among vegetables, potato, lettuce, and onion wastes are rich in polyphenols such as chlorogenic acid, caffeic acid or quercetin (Sánchez et al. 2021). Bioactive compounds can be encapsulated and used in different products namely drugs, food supplements, functional foods… (Pattnaik et al. 2021).

Moreover, it should be considered that, in many cases, the complex structure of these lignocellulosic residues requires pretreatments. These pretreatments allow the conversion of cellulose and hemicellulose into fermentable sugars and/or into bioactive compounds (Chatuverdi and Verma 2013; Patel and Sha 2021). Recent studies have demonstrated that pretreatments employing natural deep eutectic solvents, ionic liquids or hydrodynamic cavitation are interesting alternatives for this purpose (Agrawal et al. 2021; Sun et al. 2021; Sun et al. 2022). However, until now, most of the commercial-scale processes are based on acid, alkali or hydrothermal pretreatments due to their feasibility to be scaled-up and their efficiency in deconstruction of biomass (Agrawal et al. 2021; Sánchez et al. 2021). The effectiveness of these treatments is based on the breaking of bonds between carbohydrates and lignin, the reduction in crystallinity and/or an increase in the internal surface area.

When severe conditions are used for the hydrolysis processes, inhibiting compounds, which can difficult the fermentation step, can be formed (Díaz et al. 2017). These inhibitors generate physiological stress on yeast (affecting cell viability, producing a decrease of the intracellular pH or a longer lag phase) and, consequently, reducing the fermentation yield (Mahboubi et al. 2020). However, it is important to consider that some of these inhibitors, such as phenols or 5-hydroxymethylfurfural (HMF), have interesting properties as antioxidants.

Production of fermentable sugars, as well as the extraction and determination of phenolic compounds and antioxidants from fruits and vegetables have been an interesting topic in the literature (Arumugan and Manikandan 2011; Díaz et al. 2017; Tan et al. 2019; Plazzotta and Manzocco 2018; Vu et al. 2019). Nevertheless, most of these studies have been focus on

obtaining one or more value-added products (biofuels, bioactive compounds) from a specific part of a particular fruit or vegetable, i.e., peels, pulps, and seeds. Obviously, nowadays the major interest from an economic, environmental, and social perspective is to be able of using food wastes to move towards the EU circular economy action plan (CEAP 2020). With this aim in mind, in this work mixtures of overripe whole fruits and vegetables, which simulate supermarket wastes, have been used as substrates to test different hydrolysis techniques so that the obtained broths can be valorised. Two different alternatives have been considered, i.e., obtaining substrates for fermentative processes and using the wastes as source of polyphenols and antioxidants. As far as we know, the valorisation of mixed waste has scarcely been investigated (Ibarruri et al. 2021; Sahoo et al. 2021), so this approach to face real challenge is the main novelty of this work. Additionally, a whole view of valorisation possibilities for these wastes have been considered, investigating not only the production of fermentable sugars, but also the production of bioactive compounds.

2. MATERIALS AND METHODS

2.1 Raw materials

Fruits and vegetables with a degree of ripeness accurate for consumption were purchased in a local market of the north of Spain. Then, they were incubated for seven days at 25°C in order to simulate the spoilage process. After that time, the overripe fruits and vegetables were stored at 4-6°C during a maximum of a week until being treated.

Five fruits and five vegetables were selected for the experiment considering that these products represent a high percentage of total fruit and vegetable wastes generated at retail in Spain. Two different mixtures were employed:

1) A mixture of fruit constituted by 20% (w/w) of each fruit (orange, apple, pear, banana, and kiwi).
2) A mixture of vegetable constituted by 20% (w/w) of each vegetable (potato, tomato, lettuce, onion, and red pepper).

Composition of the fruits and vegetables used is shown in Table 1.

2.2 Soluble sugars

To obtain the soluble sugars, an amount of 15 g of the grinded fruit mixture, previously homogenized in a kitchen blender during 10 min, were introduced in a 250 mL Pyrex bottle and 100 mL of distilled water was added (13% w/w). In the case of vegetable wastes, in a 250 mL

Pyrex bottle 50 ml of distilled water were added to 50 g of vegetable mixture, previously homogenized in a kitchen blender (50% w/w). In both cases, after vigorously shaking the bottles, the mixture was centrifuged for 30 min at 10000 rpm (Heraeus Multifuge X1 Centrifuge Series, Thermo Fisher Scientific). The recovered supernatant was then filtered using a 20 µm cellulose filter (20 µm) and, after that, the pH was adjusted between 6 and 7 with 5M NaOH (VWR). Samples were frozen until being analysed (section 2.5).

2.3 Hydrolysis treatments

All treatments were performed in triplicate.

2.3.1 Hydrothermal hydrolysis

Samples were prepared in the same manner as described in 2.2 section and, once the bottles with fruit or vegetable wastes and water were ready, they were placed in the autoclave (AES 110 Raypa). Treatments were carried out at different temperatures (105°C, 120°C and 135°C) and at different times (5, 10, 20, 40 and 70 min). After the autoclaving processes, the content of the bottles was centrifuged during 30 min at 10000 rpm (Heraeus Multifuge X1 Centrifuge Series, Thermo Fisher Scientific) and then the supernatants were recovered and treated following the same procedure detailed in 2.2. Samples were frozen until being analysed (section 2.5).

Additionally, in case of fruit wastes, different proportions of waste:water were assayed (10, 50, 70 and 100% w/w). Samples were autoclaved at 135°C during 40 min and, after that, the supernatants were recovered and frozen until being analysed (section 2.5).

2.3.2 Acid hydrolysis

Three different acid-thermal hydrolysis were carried out. Firstly, 50 mL of 5% H_2SO_4 (Merk) (w/v) were added to 50 g of fruit waste mixture, previously homogenized in a kitchen blender, in 250 mL Pyrex bottles (50% w/w) and then the bottles were autoclaved at 135°C for 40 min. After that, solids were removed by centrifugation, supernatant was filtered and pH was neutralized to pH 6-7, as described above (section 2.2). Samples were frozen until being analysed (section 2.5).

Two additional hydrolysis was performed by means of mixing the fruit mixture with water as previously described (50% w/w) and adjusting pH to 2 and 3 with 5% sulphuric acid (Merk) before the hydrolysis process (135°C and 40 min). After that, samples were processed in the same manner detailed above.

2.3.3 Alkaline hydrolysis

For basic-thermal hydrolysis, fruit samples were prepared in the same manner as described in 2.3.2, but in this case, 50 mL of basic solution (5% (w/v) NaOH (VWR))0 were added. Additional hydrolysis were performed adjusting pH to 7, 8 and 11 with 5% NaOH (VWR) before the hydrolysis process.

2.4 Fermentation process

A mixture (9:1) of fruit and vegetable wastes (100% w/w) was hydrolysed at 135°C during 40 min and the broth obtained was employed as substrate to carry out a fermentation in Erlenmeyer flasks. *Saccharomyces cerevisiae* ACA 174 supplied by CECT (Spanish Type Culture Collection) was used and the initial microbial load was 4.10^2 CFU (Colony Former Units)/ml. Fermentation was conducted during 4 days (96 hours) in an incubator at 30°C and 50 rpm. Samples (2 g of the mixture) were taken periodically every 2 hours from the flasks, centrifuged during 10 min at 12000 rpm and the supernatant was frozen until analysing ethanol concentration and sugar content. In addition, the microbial growth was followed by means of taking 1 g of sample that was transferred to a stomacher bag and homogenized with 9 mL of sterile saline solution. After that, serial decimal dilutions of the mixture were plated in triplicate onto YPG (Yeast Extract-Peptone-Glucose) Agar Medium and incubated at 30 °C for 48 h before counting.

Fermentation was carried out in sterile conditions and in duplicate.

2.5 Analytical methods

All the analysis were carried out, at least, in triplicate.

2.5.1 Determination of total sugars: Phenol-sulphuric acid method

The content of total sugars in the samples was measured using an adaptation of the phenol-sulphuric method described by Dubois et al. (1956). For this assay, 1 mL of sample was mixed with 0.5 mL of 5% phenol and 2.5 mL of 96% H_2SO_4 (Merck). The mixture was left at room temperature for one hour and the absorbance was measured by means of a spectrophotometer (DR/2500 HACH) at 492 nm. The concentration of total sugars was determined employing glucose as standard.

2.5.2 Determination of total reducing sugars: Dinitrosalicylic acid (DNS) method.

Total reducing sugars were quantified using the Miller method (Miller 1959). In this procedure, 0.5 mL of sample was mixed with 0.5 mL of DNS reagent and the mixture was incubated at 95°C in a water bath for 5 min. After that, samples were cooled in ice and the absorbance was

measured at 540 nm using a spectrophotometer (Analytik Jena Spekol 1300/1500). Glucose was employed as standard.

2.5.3 Determination of fermentation inhibitors

Acetic acid, HMF and furfural, were analysed by HPLC (High Performance Liquid Chromatography) employing a 1200 Series model (Agilent Technologies) following the method described in Díaz et al. (2017). To quantify the concentration of acetic acid, an ICSep ICE-ION (Tecnokroma) column with a refractive index detector (RID) was used. Sulphuric acid (0.45 Mm, pH 3.1) was employed as mobile phase with a flow of 0.3 mL/min and the column temperature was 75°C. HMF and furfural were measured using a Gemini-NX 5 µm C18 110 Å column (Phenomenex) with a diode detection system (DAD). In this case, the mobile phase was methanol/water (10:90) with a flow of 1mL/min and the temperature of the column was 30°C. ChemStation software (Agilent Technologies) was employed for acquisition and analysis of data. For quantification, external analytical standards (Sigma-Aldrich) were used as reference.

2.5.4 Determination of bioethanol

A gas chromatograph CLARUS 400 (Perkin Elmer) coupled to a flame ionization detector (FID) was used for the analysis of bioethanol. The column used was an Elite WAX TR-810532 (Perkin Elmer). The column temperature was initially set at 60°C for 5 min, increased by 1°C/min to 220°C, maintaining this temperature for 40 min. The column temperature was elevated at 10°C/min to 260°C, maintaining this temperature for 10 min. Helium was used as carrier gas. The FID detector was set at 260°C. As an internal standard, 4-methyl-2-pentanol was used.

2.5.5 Determination of total phenolic compounds (TPC)

The concentration of total phenolic compounds (TPC) in the samples was determined by Folin-Ciocalteu's method (Moussi et al. 2015). In short, 3 mL of Folin-Ciocalteau 1:10 (VWR) were added to 400 µL of sample and the mixture was incubated at 22°C for 5 min. Then, 3 mL of sodium bicarbonate (6 g/100 mL) were added to the sample, and it was incubated at 22°C for 90 min. Finally, the absorbance was measured at 725 nm using a spectrophotometer (Analytik Jena Spekol 1300/1500). TPC was determined using gallic acid (Sigma Aldrich) as standard and distilled water as control.

2.5.6 Determination of antioxidant activity

The antioxidant activity of samples was determined using the method described by Moussi et al. (2015). In brief, 6 mL of 2,2-diphenyl-1-picrylhydrazyl (DPPH) solution in methanol ($6x10^{-5}$ M) (TCI) was added to 200 µL of sample. After incubation at 37°C for 20 min, the absorbance of the

mixture was measured in a spectrophotometer at 517 nm (Analytik Jena Spekol 1300/1500). Distilled water was used as control. The radical scavenging activity was determined as follows:

$$\% \text{ Inhibition: } \frac{\text{Absorbance control - Absorbance sample}}{\text{Absorbance control}} \times 100$$

2.5.7 Determination of moisture content

In order to express the results on dry weight basis (w/w), a gravimetric method was used to determine the moisture content of each batch of fruit and vegetable wastes and analysis were carried out in triplicate. Approximately 3 g of sample was weighed in a stainless-steel mortar with thick grain sea sand (Panreac). Sample and sea sand were mixed with a pestle and then the mixture was dried in an oven at 105°C for 24 hours and, after that, it was weighed again. The moisture content was calculated considering the difference between the initial and final weight of the sample.

2.6. Statistical analysis

To analyse the data, Excel software was employed to carry out a one-way ANOVA with a 95% confidence interval

3 RESULTS AND DISCUSSION

3.1. OBTAINING FERMENTABLE SUGARS

3.1.1 Soluble sugars

Just by using distilled water as extraction agent, certain amount of sugars can be extracted from fruit and vegetable wastes. Figure 1 shows results obtained from different batches of fruit and vegetable wastes obtained from a local market at different dates.

In the case of fruit residues (Figure 1a), three different batches were analysed. It should be noted that, in all cases, reducing sugars accounted for more than 50% of total sugars. According to ANOVA results, there were statistically significant differences between batches regarding both total and reducing sugars in case of fruit wastes. Despite this, it can be observed that the values of total and reducing sugars ranged between 51-59 g per 100 g of waste (dry weight) and 29-33 g per 100 g of waste (dry weight), respectively. These values were very similar to those reported in literature when fruits such as apple were analysed for sugar content. For example, Jin et al. (2019) found values of 51.5 g/100 g (dry weight) of total soluble sugars and about 35 g/100 g (dry weight) of soluble reducing sugars in apple pulp hydrolysed for 30 min at 121°C. It is necessary to keep in mind that the amount of carbohydrates depends on the kind of fruit and also on the part of the fruit. For instance, Huang et al. (2014) reported just 13 g of reducing sugars per 100 g (dry weight) from pomelo peels without hydrothermal treatment.

With respect to vegetable wastes (Figure 1b), two different batches were analysed. As in the case of fruit wastes, there were statistically differences between both total and reducing sugars content of vegetable batches employed. Again, it is remarkable that the range of variation was narrow. The amount of total (13-16 g/100 g dry weight) and reducing (10-12 g/100 g dry weight) sugars were notably lower than those obtained for fruit residues. The amount of soluble sugars in vegetable wastes is lower due to the higher presence of more complex carbohydrates. Values of total sugars of approximately 19 g/100 g (dry weight) and 20 g/100 g (dry weight), were reported for onion skin and pepper residues treated with distilled water, without hydrothermal treatment (Diaz et al. 2017; Poe et al. 2020), values slightly higher than those found here. In the case of reducing sugars, the amount extracted in this work from the unheated mixed wastes, was quite similar to that found for cabbage residues (11 g of glucose and fructose per 100 g of residues, dry weight).

As can be observed, the homogeneity of the different batches used was quite higher with respect to the moisture content (83-86% for fruit residues and 89-91% for vegetable wastes).

3.1.2 Hydrothermal treatment of fruit wastes: Effect of temperature and time

In order to hydrolyse the complex carbohydrates so that the amount of fermentable sugars extracted can be increased, different hydrothermal pretreatments have been assayed at different temperatures (105, 120 and 135°C) and times (5, 10, 20, 40 and 70 min). The amount of total and reducing sugars extracted are shown in Figure 2. Zero time indicates the amount extracted before the thermal treatment.

Even with 5 min of thermal treatment at any of the assayed temperatures, the amount of total sugars extracted were greater than that obtained in the untreated sample. For temperatures of 105 and 120°C, small differences between the samples taken at different times were observed. The amount of total sugars extracted were in all cases between 60 and 65 g/100 g dry weight. However, when substrates were subjected to the highest temperature (135°C), as the treatment time increased, the amount of total sugars also increased. After 70 min of treatment, it was possible to extract 75 g/100 g (dry weight) of fruit wastes. Oberoi et al. (2011), employed treated banana peels by hydrothermal hydrolysis at 121°C during 15 min achieving a maximum of 40 g/100 g (dry weight) for total sugars, approximately half of the amount of total sugars obtained in this work at the highest temperature (75 g/100 g). This shows the higher interest of using mixed fruit wastes that include the pulp, usually richer, in hydrolysable carbohydrates. Concerning reducing sugars, the temperature of 105°C was not high enough to hydrolyse the solubilized carbohydrates and the initial concentration of dissolved reducing sugars remained

almost constant during all the hydrothermal treatment. On the contrary, at 120°C and 135°C, it was observed an increase in the concentration of reducing sugars of 27 and 60%, respectively. In these cases, it is remarkable that, after 70 min of treatment, reducing sugars meant approximately 50% of total sugars. Statistically, the one-way ANOVA test showed a significant difference ($p < 0.05$) for broths subjected to hydrothermal treatments compared to those untreated. So, these results demonstrate the effectiveness of hydrolysis treatment in the extraction of sugars. Eroglu et al. (2017) recovered between 55 and 58 g/100 g (dry weight) of glucose and fructose by treating orange wastes at 85°C during 4 min. To obtain similar values from the fruit wastes used in this work, it was necessary to apply 135°C at least for 40 min. In this case, if theoretical values of carbohydrate content are considered, a recovery of 97% of carbohydrates is achieved.

According to ANOVA test, there were not significant differences between treating the samples, both fruit and vegetable wastes, for 40 or 70 min. So, in order to reduce costs and time, 40 min can be considered enough time for the hydrothermal treatment.

The evolution of the solid concentration (dry weight) with the time of treatment has been followed. As can be observed in figure 3, as the concentration of reducing sugars increases, the concentration of solid decreased. It is evident that higher temperature improved the solubilisation of solids and the hydrolysis of dissolved complex sugars, so that higher concentrations of reducing sugars were achieved for higher temperature of hydrolysis, as above commented. As an attempt to describe the transformations occurred in a simple way, it was developed a model based on irreversible first-order reactions (Díaz et al. 2017). It considers that solid carbohydrates are hydrolysed into soluble intermediates that then are degraded to reducing sugars. So, S is the solid matter, D is the dissolved non-reducing-sugar intermediaries and M is the dissolved reducing sugars. The parameter α is the percentage of dissolved solid matter that are carbohydrates, k_1 and k_2 are the kinetic constants of solubilisation rated of solid matter and the hydrolysis rate of soluble intermediates into reducing sugars, respectively. Model reactions are represented below.

$$\boldsymbol{S} \xrightarrow{\boldsymbol{\alpha k_1}} \boldsymbol{D} \xrightarrow{\boldsymbol{k_2}} \boldsymbol{M} \qquad \text{(Eq. 1)}$$

$$\frac{dS}{dt} = -k_1 S \qquad \text{(Eq. 2)}$$

$$\frac{dD}{dt} = \alpha k_1 S - k_2 D \qquad \text{(Eq. 3)}$$

$$\frac{dM}{dt} = k_2 D \qquad \text{(Eq. 4)}$$

Parameters were determined by fitting the data to experimental model using Microsoft Excel software. Note that only experimental data obtained after 5 min of treatment were used since during the autoclaving process the hydrolysis began before achieving the set temperature, moment considered as zero time. The comparison between experimental and model data is shown in Figure 3. The tendency is well predicted by the model for 105°C and 120°C (Fig. 3a and b), although certain discrepancies are observed for 135°C (Fig. 3c). In Table 2 are shown the values of the parameters α, k_1 and k_2. The solubilisation constant (k_1) is around ten times greater at 135ºC than for temperatures below 120ºC, whereas the hydrolysis constant is around 5 times greater for temperatures above 120ºC than for 105ºC.

3.1.3 Hydrothermal treatment of fruit wastes: Effect of fruit waste/water ratio

The highest concentration of reducing sugars achieved in the experiments showed in figure 2, was 14 g/L (135°C, 40 min). To use this broth as substrate for fermentation, it is interesting that sugar concentration is higher (Redondo et al. 2014). The addition of less water would increase the sugar concentration. However, the solubilisation process may be affected. In addition, some sugars are lost in the centrifugation process, because they remain with the separated wet solid and the amount lost is higher when the concentration is higher. For this reason and in order to maximize sugar concentration without decreasing the amount of sugar extracted per mass of fruit, different tests were carried out at 135°C during 40 min to evaluate the optimal waste/water ratio.

Figure 4 shows the amount of total and reducing sugars recovered from theses assays expressed as grams of sugar per 100 g of dry weight and grams per litre versus the percentage of fruit waste. As can be seen, with the highest addition of water (10%), the maximum quantities of both types of sugars were recovered (62 and 48 g/100 g of dry weight of waste for total and reducing sugars, respectively). When the amount of water decreases (higher percentage of fruit), the amount of sugar recovered also decreases significantly. These results suggest that the addition of water improve the extraction of sugars. Mahato et al. (2020), treated by hydrothermal hydrolysis (120°C) mixtures of fruit wastes and water (50%), recovered an amount of reducing sugars (fructose and glucose) between 12 and 50 g per 100 g (dry weight), similar values to those obtained in this work for reducing sugars when waste is added as solvent.

As expected, the highest concentration of reducing sugars (68 g/L) were found in samples without water addition (100%). It is remarkable that the sugar concentrations obtained for fruit percentages between 50% and 100%, were not very different. However, the addition of water in a 50% percentage, allowed an important increase in the amount of reducing sugars extracted. So, from a practical point of view, a 50% percentage is considered as the most suitable for the

348 hydrolysis treatment, giving broths with reducing sugar concentrations around 56 g/L. Broths
349 with similar concentration of fermentable sugars (15-60 g/L) have been used to obtain
350 bioethanol by fermentation (Arapoglou et al. 2010; Tsuji et al. 2013; Tan et al. 2019; Wilkins
351 2009).

352 It is remarkable that, in terms of concentration, there were not significant differences when it
353 was used 50, 70 and 100% proportion of fruit waste/water. Moreover, with the ratio of
354 solid:water of 50% it was possible to extract an adequate amount of reducing sugars, so from a
355 practical point of view, it could be considering enough proportion for the hydrolysis treatment.

356 #### 3.1.4 Hydrothermal treatment of fruit waste: Effect of pH

357 It is well known that pH value importantly influences over the efficacy of hydrolysis processes
358 (Rodriguez-Valderrama et al. 2020; Kim et al. 2013). For this reason, acid and basic hydrolysis
359 have been tested in order to maximize the amount of sugars that can be obtained from fruit
360 residues. Figure 5 shows the amount of total and reducing sugars obtained in samples before
361 and after being treated at 135°C for 40 min for different pH values.

362 Regarding acid treatments, the maximum content of both types of sugars were obtained after
363 hydrothermal hydrolysis at pH 3 (35 and 28 g per 100 g (dry weight) of fruit wastes for total and
364 reducing sugars, respectively). This result is very similar to that obtained at pH 2. So, slight acid
365 pH values improve the sugar extraction and the hydrolysis of carbohydrates. It has been
366 reported that the use of acids for pretreatment of lignocellulosic structures favour the
367 solubilization of hemicellulose, making it more available for subsequent treatments (Rodriguez
368 et al. 2017). Nevertheless, when a 5% H_2SO_4 acid solution was employed as extracting solvent
369 (pH 1.3), the amount of sugar obtained decreased, being the values obtained after treatment
370 lower than those obtained before. Concerning alkaline pretreatments, as the pH in the samples
371 increased, the amount of sugars extracted decreased with values lowers than those obtained in
372 the pH range 3-5.

373 These results are in accordance with those obtained in the literature. For instance, Widmer et
374 al. (2010) studied the effect of the pH (3 and 7) in the extraction of fermentable sugars from
375 orange wastes and the best results were obtained in broths with pH adjusted to 3 (22 of glucose
376 and fructose/100 g dry weight). However, when the pH of the samples was adjusted to 7, the
377 amount of reducing sugars decreased to 18 g/100 g (dry weight). These values are quite similar
378 to those obtained in this work for neutral pH. Moreover, Suhag et al. (2020) comparing acid and
379 basic hydrolysis of banana waste to obtain reducing fermentable sugars, observed a similar

tendency that in this work with better results with acid than with alkaline pretreatment (25 and 30 g per 100 g of dry weight for basic and acid hydrolysis, respectively).

3.1.5. Hydrothermal pretreatment of vegetable wastes

The production of vegetable wastes at retail level is usually lower than the production of fruit wastes. For example, in Spain the vegetable wastes generated by supermarkets is less than 10% the fruit wastes. So, considering that both kind of wastes could be treated in the same facility, the conditions selected as many suitable for fruit wastes have been tested for vegetable wastes.

Figure 6A shows the amount of total and reducing sugars recovered from vegetable wastes mixed with water (50% w/w) and treated by thermal hydrolysis at 135°C for different times (5,10,20 and 40 min). As can be seen in Figure 6a, the amount of total and reducing sugars obtained after 40 min of treatment were 23 and 13 g per 100 g (dry weight), respectively. These values were lower than those obtained from fruit residues (32 and 22 g/100 dry weight). In the case of reducing sugars, the amount extracted slightly increased with treatment time and values obtained after treatment were similar to those obtained in samples without treatment (10-12 g/100 g dry weight). Diaz et al. (2017) obtained almost the same amount of reducing sugars (14-18 g/100 g dry weight) from tomato and pepper wastes employing 110°C and 5 min of treatment. However, the total sugars doubled their concentration after 20 min of treatment. According to theoretical values, hydrothermal pretreatment at 135°C for 40 min resulted in the recovery of 41% of total carbohydrates.

The evolution of the solid concentration (dry weight) with the time of treatment has also been followed for vegetables wastes, observing a similar behaviour than that observed with the fruit wastes (Fig. 6b). The model above commented (Eq. 1-4) was used to fit the experimental data with good results even for the shortest times of treatment. As shown in Table 2, the solubilisation constants at 135ºC are very similar for fruit and vegetable wastes, whereas the hydrolysis constant is more than 2 times greater for vegetable wastes.

As in the case of fruit residues, tests were carried out at different ratios of waste/water (10, 50 and 100%, w/v, of vegetable wastes) at 135°C and 40 min of treatment. As it is shown in Table 3, the best recovery results were obtained with a percentage of waste of 10%: 52 and 42 g per 100 g of total and reducing sugars (dry weight), respectively. Martin-Lara et al. (2020) using pepper waste as feedstock and a ratio of 10% (residue/water) recovered less glucose (25 g/100 g dry weight) than in this work. As it was expected, in terms of concentration the highest values of reducing sugars (30 g/L) were obtained in broths without water addition (100%).

Again, as the proportion of vegetable wastes in relation with water increased, the content of total and reducing sugars that can be extracted decreased. This is due to the fact that, during the hydrothermal hydrolysis process, the less water there is, the more difficult it will be to extract sugars from the solid waste. A greater water/solid ratio generates a greater driving force, which means a better diffusion of the compounds to the liquid medium. This favors the immersion of the fermentable sugars to be extracted in the water, which facilitates their extraction (De Paula et al.2021; Caldas et al. 2018).

3.2. HYDROLYSATE FROM FRUIT AND VEGETABLE WASTES AS SUBSTRATE FOR FERMENTATION.

During the hydrothermal hydrolysis some fermentation inhibitors such as acetic acid, furfural and HMF could be formed. So, the concentration of these inhibitory compounds was analysed in all broths obtained after the hydrolysis treatments. For acetic acid, 6 g/L has been reported inhibitory concentrations of fermentative microorganisms (Zheng et al. 2013). In addition, for HMF and furfural, concentrations of 5 and 1 g/L, respectively, have been reported to inhibit fermentation process (Lee and Jeffries 2011). In the case of acetic acid, for all treatment tested, the concentration of this inhibitor in the samples was below the detection limit (< 1 mg/L). Regarding HMF and furfural, concentrations much lower than those describe as inhibitory in the literature were detected: 0.025-1 and 0.008-0.2 g/L for HMF and furfural, respectively (Table 4). The presence of these inhibitors could affect the fermentation yield and productivity because of the stress generated on yeast as indicated in introduction section.

According to results described previously a mixture of fruit (90%) and vegetable (10%) wastes (100% w/w) was selected to be fermented after being hydrolized at 135°C for 40 min. As example of fermentation, the hydrolysate was fermented employing *Saccharomyces cerevisiae* in order to produce bioethanol. The resuls obtained from fermentation, i.e., ethanol production, evolution of reducing sugars and yeast's growth are shown in figure 7.

As can be seen in Figure 7, with an initial reducing sugars concentration of 53 g/L a maximum value of 27 g/L of ethanol was obtained after 96 hours of fermentation. The yield of ethanol production with respect to substrate consumption was 0.51 g ethanol/g sugar, very near the theorical value. The highest rate of ethanol production occurred between 12 and 24 hours with a specific ethanol productivity of around 2 g/Lh. During the fermentation process, yeast increased from 10^2 to 10^4 CFU/ml achieving a maximum concentration after 88 hours of incubation.

Very similar values of ethanol concentration and productivity were found in the literature when similar substrates were used. For example, Singh et al. (2011), obtained an ethanol

concentration and ethanol productivity of 28,2 g/L and 2.3 g/Lh respectively from banana peel hydrolysates using *S. cerevisiae* as fermentative microorganism. Chohan et al. (2020) using potato peel hydrolysates as fermentation substrate, achieved a maximum bioethanol concentration of 23 g/L with an ethanol productivity of 1.5 g/L/h. According to theses results, fruit and vegetable wastes could be considered an adequate substrate for fermentation processes to obtain bioethanol on other bioproducts of interest.

3.3. OBTAINING BIOACTIVE COMPOUNDS

3.3.1. Antioxidant capacity and TPC: Effect of temperature and time.

The TPC and antioxidant properties both obtained from fruit and vegetable residues subjected to different hydrolytic treatments were determined and results are shown in figure 8.

For fruit and vegetable residues treated at different temperatures and times (Figure 8a and 8b), the maximum antioxidant activity was obtained in broths treated at 120°C for 40 min with the initial pH of the medium adjusted to 8 (73 and 63 % of DPPH inhibition for fruit and vegetable residues, respectively). In all tests carried out, the hydrolysis treatment gave an increase in the antioxidant capacity of the broths with respect to the results obtained in samples without treatment. Some authors have reported values of antioxidant activity from orange and onion wastes of approximately 70 and 60% respectively (Annu et al. 2018; Nile et al. 2018), similar to those obtained here in fruit and vegetable broths just after 10 min of treatment.

Concerning phenolic content, in broths from fruit wastes (Figure 8a), it can be observed that as treatment time increased, the extracted TPC also increased for all temperatures tested. It is surprising that the maximum concentration of TPC (8 mg gallic acid equivalent (GAE)/g) was obtained at 135°C, which does not match with the maximum DPPH inhibition. Guthrie et al. (2020) obtained approximately 15 mg GAE /g (dry weight) from kiwifruit wastes heated at 120°C during 30 min of treatment, almost double TPC than that obtained in the present work at similar conditions. At elevated temperature, the diffusivity and solubility of phenolic compounds increase improving the extraction (Liu et al. 2014), which would explain that for higher temperatures, the amount of TPC extracted in this work is higher.

The maximum TPC measured in broths from vegetable wastes (Figure 8b), was 2 mg GAE/g (dry weight), much lower than in the case of fruit wastes. It is noticeable that the percentage of DPPH inhibition is quite similar in broths from fruit and vegetable wastes, whereas in the TPC there are important differences for TPC. Phenolic compounds, especially phenolic acids, and flavonoids are commonly found in fruit residues. However, in vegetable wastes the antioxidant

capacity is mainly due to other non-phenolic compounds such as carotenoids present in tomato or pepper (Kabir et al. 2015; Marcillo-Parra et al. 2021).

3.3.2. Antioxidant activity and TPC: Effect of pH

Figure 8c shows results of hydrolysis of fruit wastes carried out at different pH values. Although differences were quite low, the highest antioxidant activity was obtained after hydrolysis at pH 8 (Figure 8c). In accordance with these results, it has been reported that higher pH values increase the antioxidant capacity of the substrate, probably due to a faster extraction of polyphenols and polysaccharides (Mellinas et al. 2020). For TPC results differences obtained at different pH values are more abrupt. So, as the pH of fruit broths increased, the phenolic content was higher (Figure 8C), obtaining a maximum of 12 mg GAE/g /dry weight) at pH 11.

Lafka et al. (2011) studied the effect of pH (2-6) on the extraction of phenolic content in olive wastes. The results showed that, as the pH of the medium increases, less phenolic compounds were obtained, an opposite trend to that obtained in this work, where the maximum amount of TPC was obtained at basic pH, in accordance with the higher extraction of polyphenols at higher pH values reported by Mellinas et al. (2020).

3.3.3. Antioxidant capacity and TPC: Effect of waste/water ratio

The highest antioxidant activities (Figure 8d), for both fruit and vegetable residues, were obtained without water addition (100% of waste): 92 and 66 % DPPH inhibition for fruit and vegetable residues, respectively. Results showed notable differences between broths from fruit and vegetable residues for the percentages 10% and 100%. On the contrary, when 50% is used the antioxidant capacity obtained is almost the same. Arab et al. (2019), reported similar values (30-35% of DPPH inhibition) in broths from tomato wastes in a proportion of 10%, that those obtained in this work from vegetable wastes with the same percentage of waste in water. Feumba et al. (2020) obtained a percentage of DPPH inhibition between 10 and 40% from mango, orange, apple, and banana wastes with a percentage 10% of wastes, which are values much lower than those obtained here from fruit wastes.

Figure 8d shows the amount of phenolic compounds obtained from the tests carried out with different residue/water ratio. As can be observed, TPC increases when the addition of wastes increases. This behaviour is related to the mass transfer principle, according to which it is greater when a higher solvent/solid ratio is used (Al-Farsi and Lee 2008). For all waste to water ratio tested, the amount of total phenolic compounds in fruit was higher than in vegetable wastes

with a maximum value obtained with the percentage 10% in both cases: 9.5 and 8.5 mg GAE /g dry weight, respectively.

Tunchaiyaphum et al. (2013), evaluated the effect of various solid:water proportions (10, 20, 30, 40 and 50%) on phenolic compounds recovery from mango peels. They observed that, as in the case of this work, the best results were obtained with the greatest proportion of water: 40 mg GAE per gram (dry weight), almost 4 times more than those obtained here with the highest percentage of waste (10%). Saleem and Saeed (2019), reported values between 15-25 mg GAE /g (dry weight) for orange, banana and lemon peel in a proportion of 10% of waste/water and without heat treatment. These values are much higher than those obtained in this work for all ratios tested since citrus fruits are characterized by a high content of phenolic compounds (Multari et al. 2020; Dong et al. 2019) and the mixture used here only included 20% of citrus fruits (oranges).

The best results obtained in the present work are comparable with those reported by other authors who also investigated the use of fruit and vegetable wastes to obtain different compounds. Table 5 shows an overview of the best results found here compared with literature data. It can be seen that the use of a mixture of whole fruits allows to obtain similar, or even higher, values of reducing sugars and TPC, and also higher antioxidant activity, than those reported in other works carried out with peels or seeds. Hence, fruit and vegetable wastes (FVW) are an interesting substrate for the obtention of bioactive compounds and fermentable sugars.

4. CONCLUSION

A hydrothermal treatment of fruit or vegetable wastes at 135°C for 40 min increased the amount of reducing sugars extracted around 30% with respect to the absence of treatment. The ratio waste:water was an important factor because the use of more water increased the amount of reducing sugars extracted but also implied a dilution of sugars concentration. With respect to pH values, the best results were obtained in the range 2-5. The kinetic constants for the solubilisation and hydrolysis processes were estimated with values in the ranges 0.002-0.02 min^{-1} and 0.0006-0.008 min^{-1}, respectively. Broths, obtained by fermenting a mixture of fruit and vegetables treated at 135° C and 40 min that contained around 56 g/L of reducing sugars and low concentrations of inhibitors, were directly used as fermentation media giving a product with an ethanol concentration of 27 g/L. In addition, it was observed that the hydrothermal treatment increased the TPC and the antioxidant capacity of the broths revealing this kind of wastes as interesting sources of antioxidants and polyphenols. The highest antioxidant activities were obtained after submitting the wastes at 135°C and 40 min without adding water; the values

obtained were 92 and 66% of DPPH inhibition for fruit and vegetable wastes, respectively. For phenolic content, the maximum amount was extracted from fruit residues at pH 11, obtaining 12 mg GAE/g (dry weight). The results obtained in this work remarked the versatility and potential interest of theses wastes in order to employ them as substrates for different applications in biotechnological, food and pharmaceutical sectors. Although significant advances have been achieved in the exploitation of fruit and vegetable wastes as a source of high value-added products, this field requires interdisciplinary research from food chemistry, engineering, and biotechnology areas. The use of FVW relies on three future proposals: 1) the optimization of pretreatment techniques for wastes to be employed in fermentative processes, 2) development of innovative applications of bioactive compounds in different products and 3) implementation of efficient and cost-effective procedures for obtaining value-added products.

5. STATEMENTS AND DECLARATIONS

Ethics approval and consent to participate

Not applicable.

Consent for publication

Not applicable.

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Competing interests

The authors declare that they have no competing interest.

Authors' contributions

All authors contributed to the study conception and design. Conceptualization, investigation, data curation and formal analysis were performed by Marta Sánchez, Amanda Laca and Adriana Laca. The first draft of the manuscript was written by Marta Sánchez. The revision and edition of the manuscript were performed by Amanda Laca and Adriana Laca. Funding acquisition and supervision were performed by Mario Diaz. All authors read and approved the final manuscript.

Availability of data and materials

All data generated or analyzed during this study are included in this published article.

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818

819

820 **FIGURE CAPTIONS**

821 Fig.1 Soluble total (A) and reducing (B) sugars extracted from different batches of fruit and vegetable
822 wastes. Fruit wastes were added to water in a percentage of 13% and vegetable wastes in a percentage
823 of 50% (w/w)

824 Fig.2 Total and reducing sugars obtained from the fruit residues (fruit wastes added in a percentage of
825 13% (w/w)) subjected at hydrolysis treatments at different temperatures: 105, 120 and 135°C and pH 4.5

826 Fig.3 Evolution of solubilized reducing sugars solubilized non-reducing-sugar intermediates, total sugars
827 and reducing sugars in solid phase with time for different temperatures tested: 105°C (A), 120°C (B) and
828 135°C (C). Symbols correspond to experimental data and lines correspond to model results

829 Fig.4 Total and reducing sugar content extracted from fruit residues with different proportion of
830 waste:water: 10, 50, 70 and 100 % (w/w) at 135°C during 40 min and pH 4.5

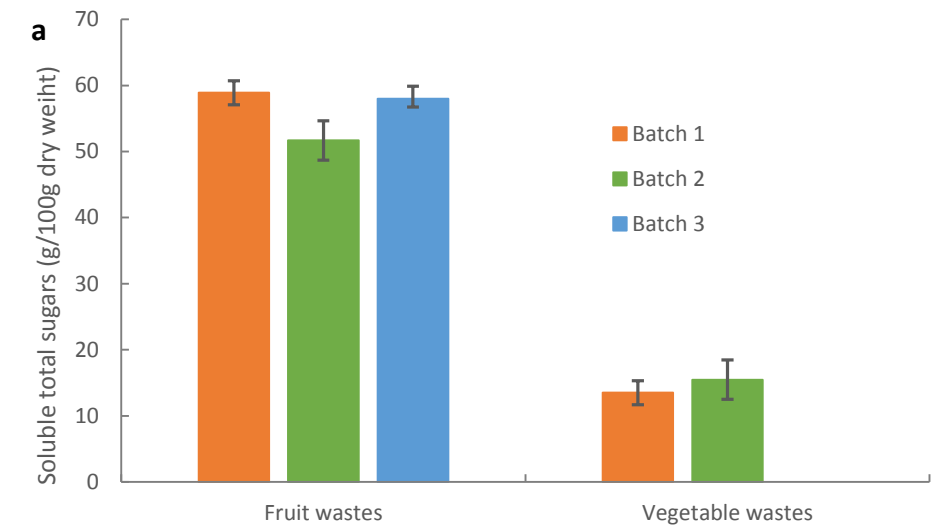
831 Fig.5 Total and reducing sugars extracted from fruit wastes (50% of waste) before and after acid and alkali
832 hydrolysis pretreatments (135°C, 40 min)

833 Fig.6 (A) Total and reducing sugar obtained from vegetable wastes (vegetable wastes added in a
834 percentage of 50% (w/w)) subjected at hydrothermal treatment at 135°C and pH 5 and (B) evolution of
835 solubilized reducing sugars, solubilized non-reducing-sugar intermediates and reducing sugars in solid
836 phase with time. Symbols correspond to experimental data and lines correspond to model results

837 Fig.7 Ethanol production, reducing sugar consumption and yeast growth during the fermentation process
838 of a mixture of fruit and vegetable wastes treated at 135°C during 40 min and pH 4.5

839 Fig.8 (A) Antioxidant capacity and TPC (unfilled) of broths from fruit wastes (13% of waste, w/w) at
840 different temperatures (105ºC, 120°C, 135°C and 120°C pH 8) and times; (B) Antioxidant capacity and TPC
841 of broths from vegetable wastes (50% of waste, w/w) at different temperatures (120°C, 135°C and 120°C
842 pH 8) and times; (C) Antioxidant capacity and TPC in fruit residues after 135°C and 40 min of treatment
843 and different pH; (D) Antioxidant capacity and TPC of broths from fruit (filled) and vegetables (unfilled)
844 wastes treated at 135°C during 40 min of treatment with different percentage of wastes: 10, 50 and 100%

Fig1


a
Soluble total sugars (g/100g dry weiht)
70
60
50
40
30
20
10
0
Batch 1
Batch 2
Batch 3
Fruit wastes
Vegetable wastes

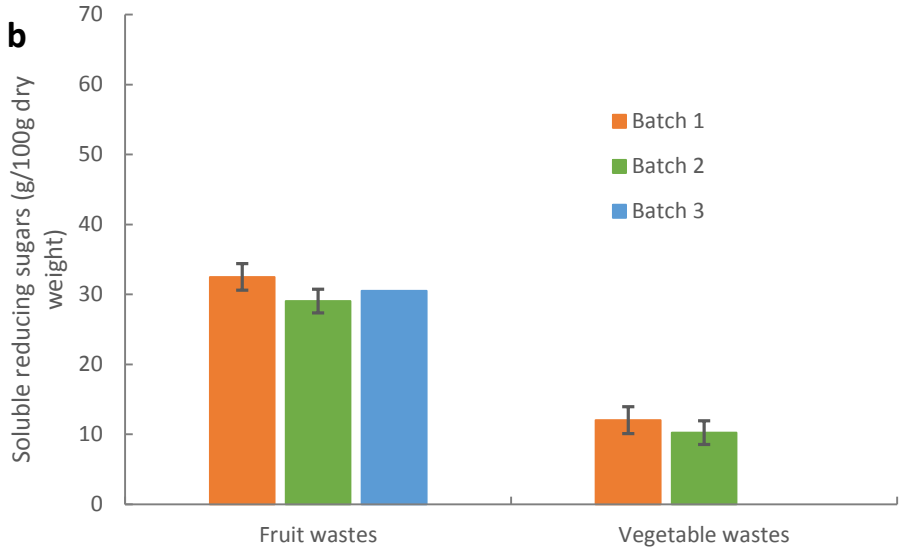

b
Soluble reducing sugars (g/100g dry weight)
70
60
50
40
30
20
10
0
Batch 1
Batch 2
Batch 3
Fruit wastes
Vegetable wastes

Fig2

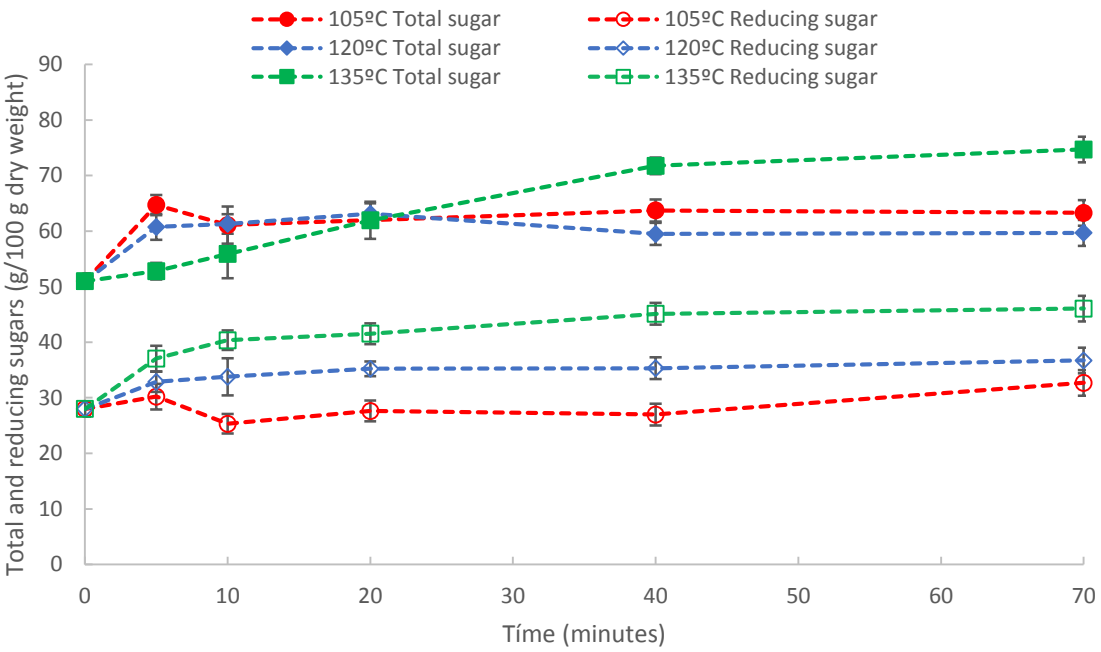

105ºC Total sugar
105ºC Reducing sugar
120ºC Total sugar
120ºC Reducing sugar
135ºC Total sugar
135ºC Reducing sugar
Total and reducing sugars (g/100 g dry weight)
0
10
20
30
40
50
60
70
80
90
0
10
20
30
40
50
60
70
Time (minutes)

Fig3

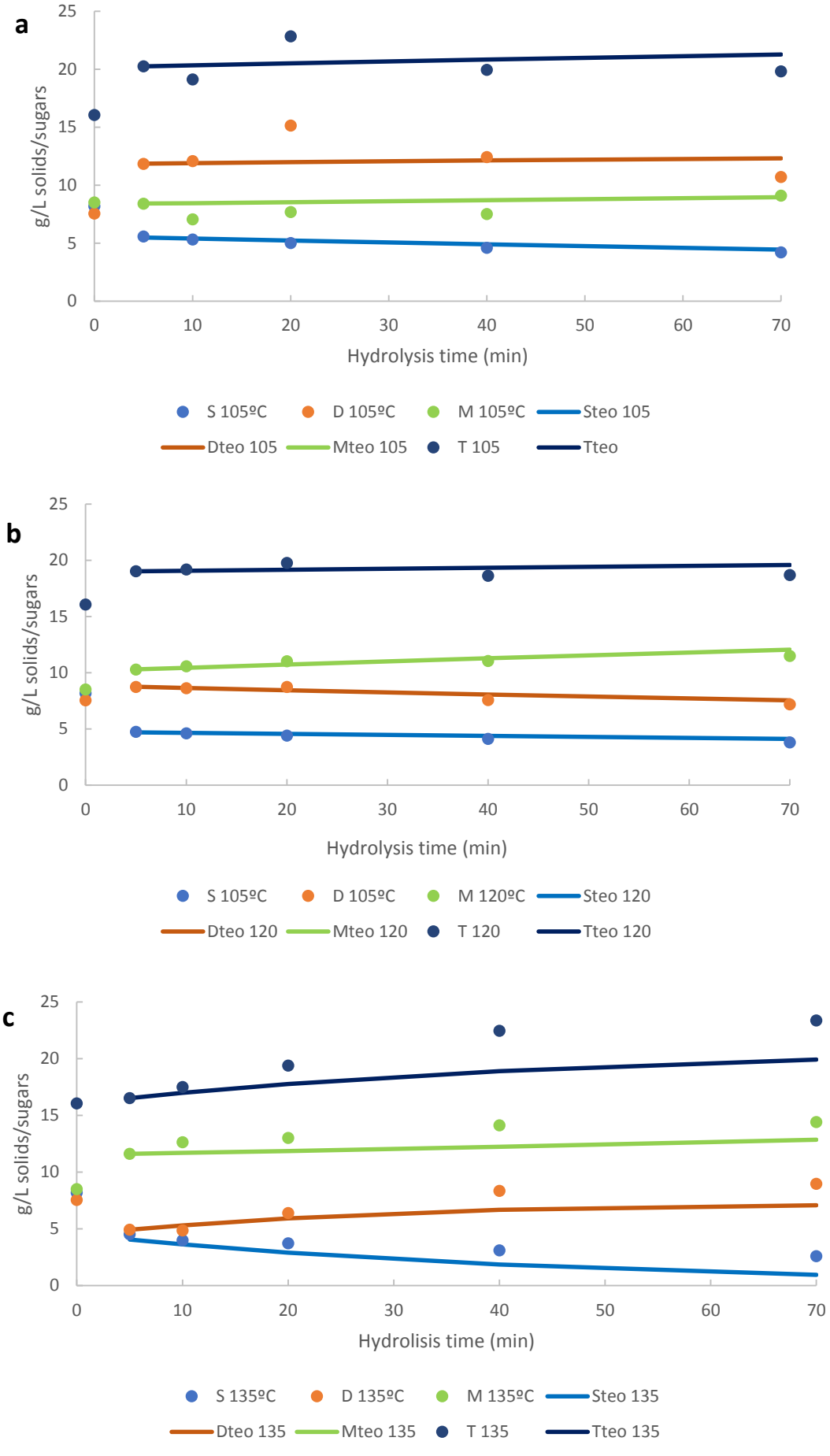
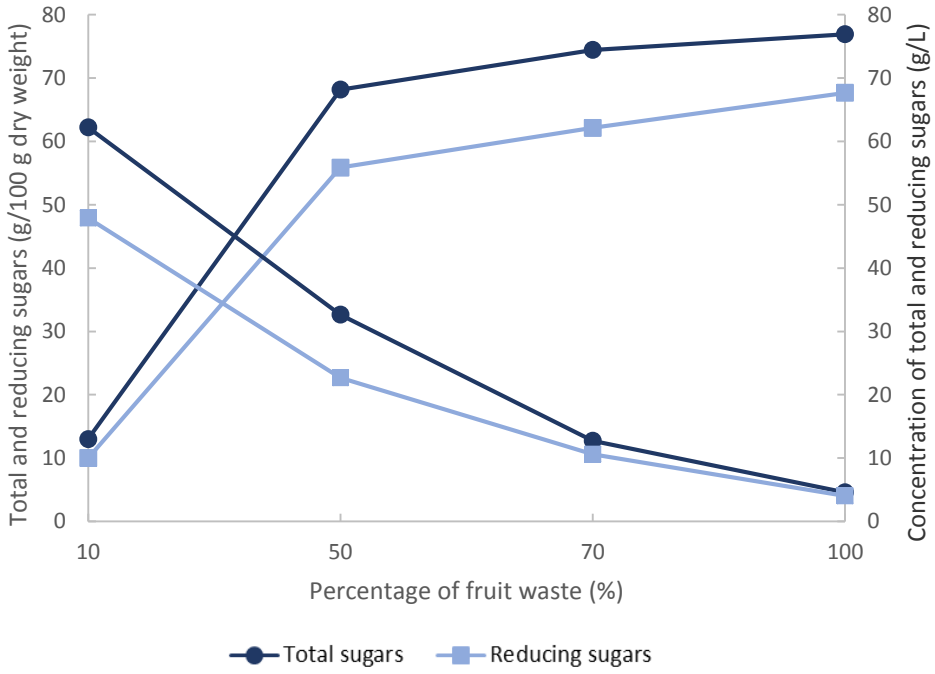

a
g/L solids/sugars
Hydrolysis time (min)
S 105ºC
D 105ºC
M 105ºC
Steo 105
Dteo 105
Mteo 105
T 105
Tteo
b
g/L solids/sugars
Hydrolysis time (min)
S 105ºC
D 105ºC
M 120ºC
Steo 120
Dteo 120
Mteo 120
T 120
Tteo 120
c
g/L solids/sugars
Hydrolisis time (min)
S 135ºC
D 135ºC
M 135ºC
Steo 135
Dteo 135
Mteo 135
T 135
Tteo 135

Fig4



Total sugars
Reducing sugars

Fig5

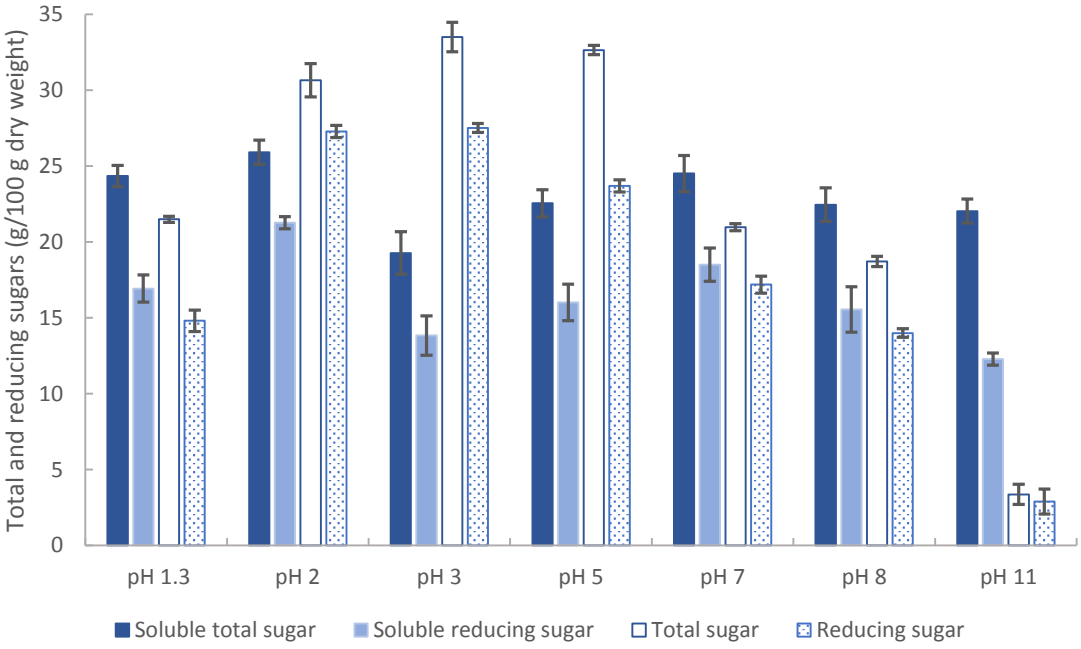
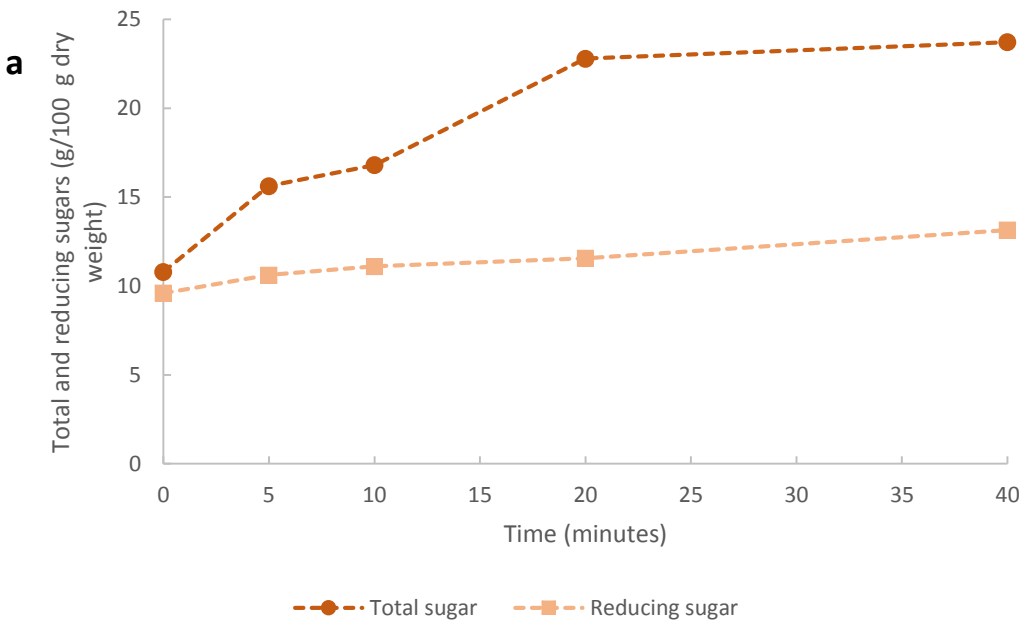

Total and reducing sugars (g/100 g dry weight)
35
30
25
20
15
10
5
0
pH 1.3
pH 2
pH 3
pH 5
pH 7
pH 8
pH 11
Soluble total sugar
Soluble reducing sugar
Total sugar
Reducing sugar

Fig6


a
Total and reducing sugars (g/100 g dry weight)
25
20
15
10
5
0
0
5
10
15
20
25
30
35
40
Time (minutes)
Total sugar
Reducing sugar

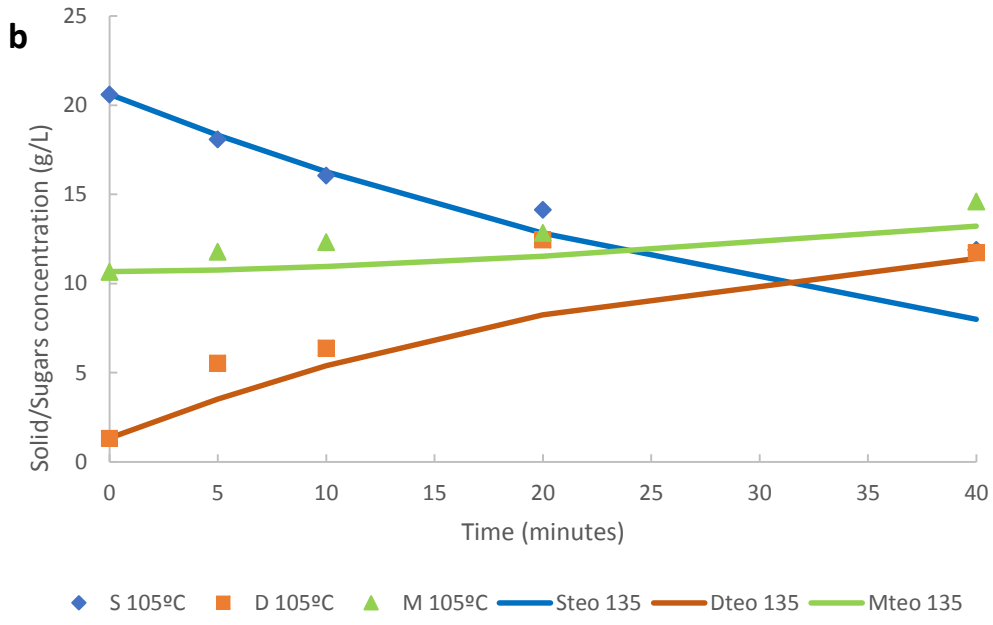

b
Solid/Sugars concentration (g/L)
25
20
15
10
5
0
0
5
10
15
20
25
30
35
40
Time (minutes)
S 105ºC
D 105ºC
M 105ºC
Steo 135
Dteo 135
Mteo 135

Fig7

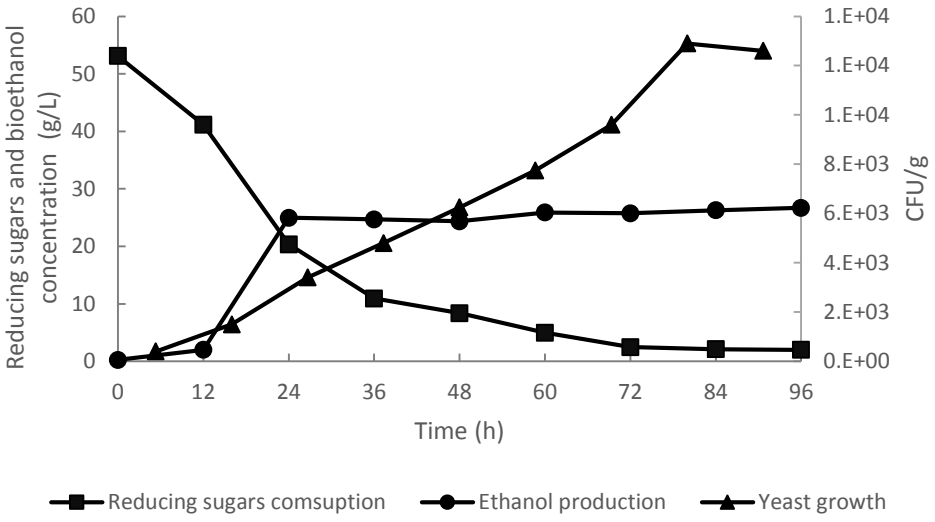
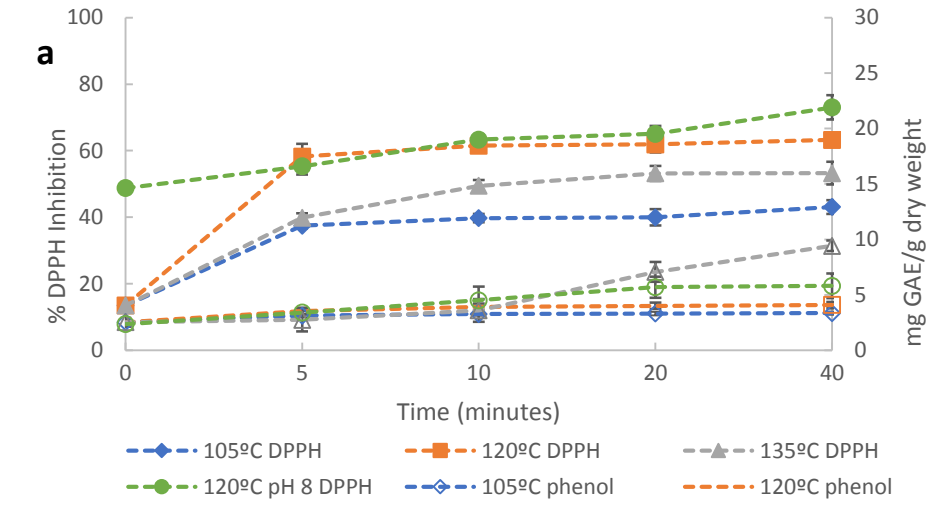
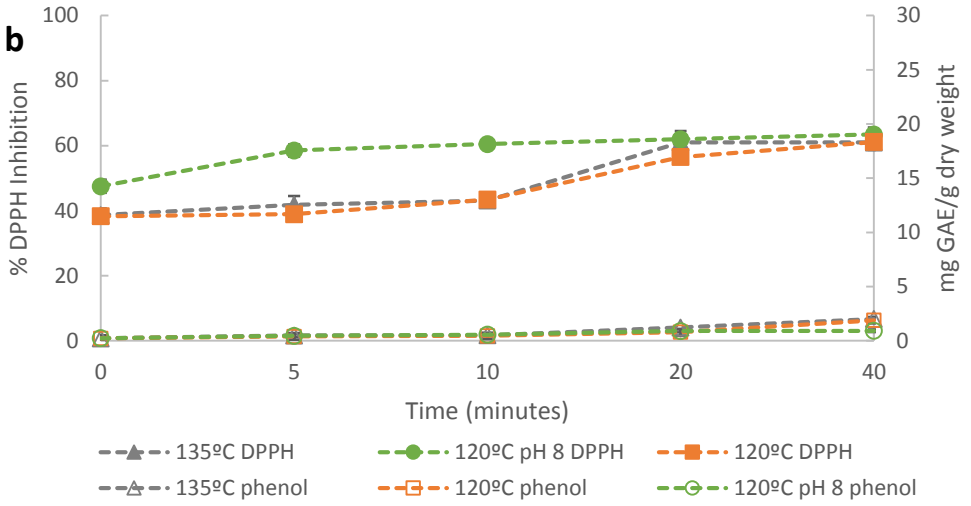
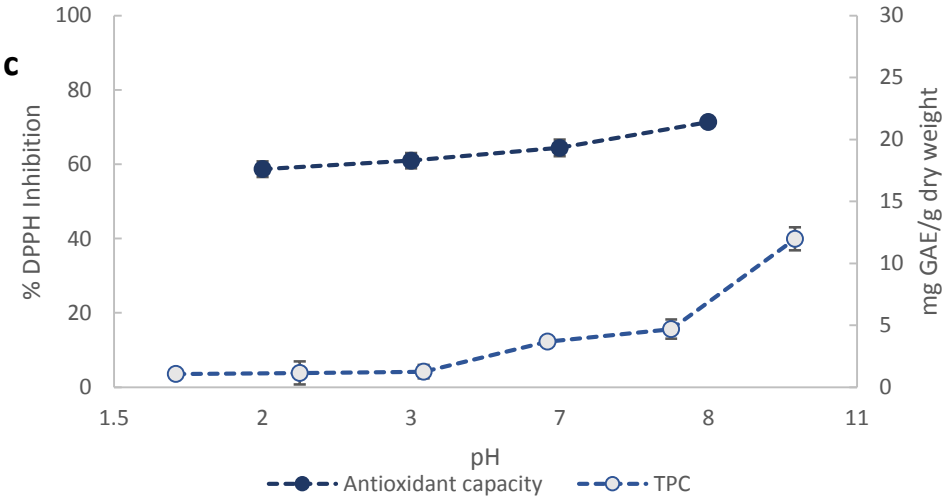

Reducing sugars and bioethanol concentration (g/L)
60
50
40
30
20
10
0
1.E+04
1.E+04
1.E+04
8.E+03
6.E+03
4.E+03
2.E+03
0.E+00
CFU/g
0
12
24
36
48
60
72
84
96
Time (h)
Reducing sugars comsuption
Ethanol production
Yeast growth

Fig8


a
% DPPH Inhibition
mg GAE/g dry weight
Time (minutes)
105ºC DPPH
120ºC DPPH
135ºC DPPH
120ºC pH 8 DPPH
105ºC phenol
120ºC phenol


b
% DPPH Inhibition
mg GAE/g dry weight
Time (minutes)
135ºC DPPH
120ºC pH 8 DPPH
120ºC DPPH
135ºC phenol
120ºC phenol
120ºC pH 8 phenol


c
% DPPH Inhibition
mg GAE/g dry weight
pH
Antioxidant capacity
TPC

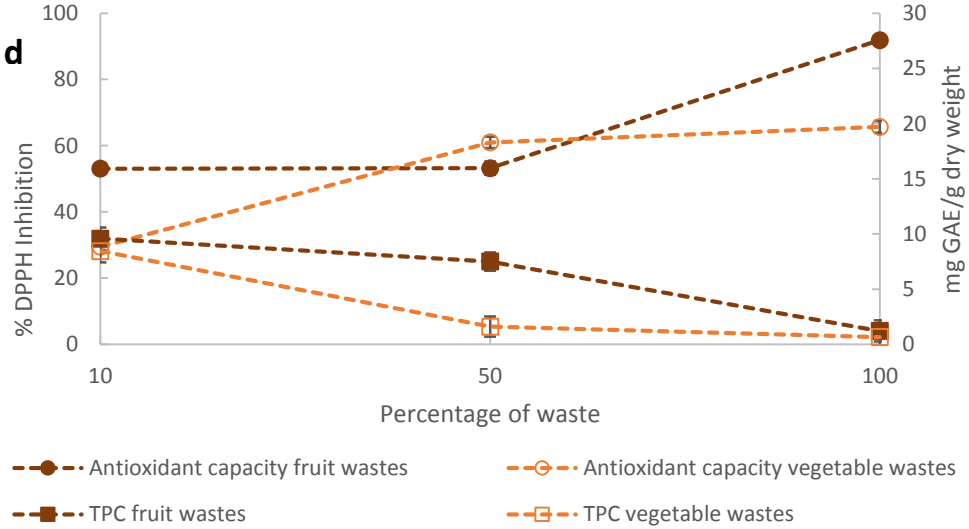

d
% DPPH Inhibition
mg GAE/g dry weight
Percentage of waste
Antioxidant capacity fruit wastes
Antioxidant capacity vegetable wastes
TPC fruit wastes
TPC vegetable wastes

Table 1

Table 1. Nutritional composition of the fruits and vegetables used as substrate (g/100 g edible portion).

	Carbohydrates[a] (g)	Fibre[a] (g)	Lipids[a] (g)	Total protein[a] (g)	Vitamins[a] (mg)	Minerals[a] (mg)	Moisture[b] (g)
Orange	8.6	2	Trace	0.8	50[A]	280[A]	78.6
Apple	12	2	Trace	0.3	4[B]	122[B]	87.9
Pear	10.6	2.3	Trace	0.4	3[C]	169[C]	81.6
Banana	20	3.4	0.3	1.2	11[D]	427[D]	82.5
Kiwi fruit	10.6	1.9	0.5	1.1	59.1[E]	368.5[E]	86.3
Tomato	3.5	1.1	0.1	0.9	20.1[F]	298[F]	95.7
Potato	15.2	1.7	0.2	2.2	19.3[G]	618[G]	78.3
Pepper	4.5	1.8	0.6	1.3	153.2[H]	193.6[H]	93.2
Lettuce	1.4	1.5	0.6	1.1	13[I]	295.6[I]	96.5
Onion	5.3	1.8	Trace	1.1	7.5[J]	228.3[J]	94.9

[a] Values from Base de Datos Española de Composición de Alimentos (BEDCA)

[b] Own data (average values)

[A] Vitamins (A, E B_6, C) / Minerals (Ca, Fe, K, Mg, Na, P, Se, Zn, I^-)

[B] Vitamins (A, E, B_6, C) / Minerals (Ca, Fe, K, Mg, Na, P, Se, Zn)

[C] Vitamins (A, B_6, C) / Minerals (Ca, Fe, K, Mg, Na, P, Zn, I^-)

[D] Vitamins (A, E, B_6, C) / Minerals (Ca, Fe, K, Mg, Na, P, Se, Zn, I-)

[E] Vitamins (A, E, B6, C) / Minerals (Ca, Fe, K, Mg, Na, P, Se, Zn, I-)

[F] Vitamins (A, E, B6, C) / Minerals (Ca, Fe, K, Mg, Na, P, Se, Zn, I-)

[G] Vitamins (B_6, C) / Minerals (Ca, Fe, K, Mg, Na, P, Se, Zn, I-)

[H] Vitamins (A, E, B6, C) / Minerals (Ca, Fe, K, Mg, Na, P, Se, Zn, I-)

[I] Vitamins (A, E, B6, C) / Minerals (Ca, Fe, K, Mg, Na, P, Se, Zn, I-)

[J] Vitamins (E, B6, C) / Minerals (Ca, Fe, K, Mg, Na, P, Se, Zn, I-)

Table 2. Kinetic constants calculated from experimental data

Kind of wastes	Temperature (ºC)	α	k_1 (min^{-1})	k_2 (min^{-1})
FRUIT	105	0.95	0.00320	0.00063
	120	0.95	0.00203	0.00332
	135	0.95	0.02242	0.00298
VEGETABLE	135	1	0.02371	0.00837

Table 3

Table 3. Content of total and reducing sugars obtained from test carried out at different w/v proportion of vegetable wastes at 135°C during 40 min and pH 5, expressed in grams per 100 grams of dry weight.

% w/v	Total sugar	Reducing sugar
10	51.98±0.88	42.43±0.26
50	23.72±1.23	13.15±0.61
100	3.71±0.98	3.37±1.25

Table 4. Concentration of inhibitors (acetic acid, HMF, furfural) of broths obtained from different hydrolysis pretreatments.

FRUIT WASTES			
Hydrolysis condition	**Acetic acid (g/L)**	**HMF (g/L)**	**Furfural (g/L)**
105°C 5,10,20,40,70 min	nd	nd	nd
120°C 5,10,20,40,70 min	nd	nd	nd
135°C 5,10,20,40,70 min	nd	0.009-0.15	0.008-0.005
10% (w/v)	nd	0.04	0.009
50% (w/v)	nd	0.5	0.02
70% (w/v)	nd	0.6	0.02
100% (w/v)	nd	1	0.03
pH 2	nd	0.2	nd
pH 3	nd	0.15	nd
pH 7	nd	0.07	nd
pH 8	nd	0.08	nd
pH 11	nd	0.05	nd
VEGETABLE WASTES			
Hydrolysis condition	**Acetic acid (g/L)**	**HMF (g/L)**	**Furfural (g/L)**
135°C 5 min	nd	0.005	nd
135°C 10min	nd	0.005	nd
135°C 20 min	nd	0.007	nd
135°C 40 min	nd	0.02	nd
10% (w/v)	nd	0.003	nd
100% (w(v)	nd	0.13	nd

nd. Non Detected

Table 5

Table 5. Comparison of sugar content and bioactive compound extraction between different literature works and this study.

Waste	Treatment	Compound	Amount	Reference
Mixture of fruit wastes	Hydrolysis	Reducing sugars	68 g/L	Present work
Mixture of fruit wastes	Hydrolysis	Antioxidant activity	92% DPPH Inhibition	Present work
Mixture of fruit wastes	Alkali hydrolysis	TPC	12 mg GAE/ g dw	Present work
Mixture of fruit wastes	Hydrolysis	Reducing sugars	76 g/L	Zanivan et al. 2022
Apple peel	Water extraction	Reducing sugars	67.3 g/L	Tempelman et al. 2021
Onion skin	Alkali extraction	Reducing sugars	60 g/L	Blue et al. 2021
Pomegranate peel	Acid hydrolysis	Reducing sugars	56 g/L	Saleem et al. 2022
Grape seed	Organic solvent extraction	Antioxidant activity	60% DPPH Inhibition	Akca and Akpinar, 2021
Onion skin	Organic solvent extraction	Antioxidant activity	82% DPPH Inhibition	Nile et al. 2018
Mango peel	Organic solvent extraction	Antioxidant activity	96% DPPH Inhibition	Feumba et al. 2020
Mango peel	Organic solvent extraction	Antioxidant activity	56% DPPH Inhibition	Marcillo-Parra et al. 2021
Kiwifruit peel	Subcritical water extraction	TPC	20 mg GAE/g dw	Guthrie et al. 2020
Garlic husk	Organic solvent extraction	TPC	11.80 mg GAE/g dw	Kallel et al. 2014
Lemon peel	Organic solvent extraction	TPC	8 mg GAE/g dw	Dong et al. 2019
Citrus peel	Water extraction	TPC	6 mg GAE/g dw	Gómez-Mejía et al. 2019