PHYSICOCHEMICAL CHARACTERIZATION OF WASTE FROM PROCESSING OF FRUIT PULP: ONE DISCUSSION ABOUT SOME PROCESSES FOR BIOPRODUCTS RECOVERY

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ABSTRACT

Brazil is the third major producer of fruits in the world and, as a result of requirement for preservation and processing, generate large amounts of waste, such as bark, seeds and filter cake (waste pulp) and the sustainability of the process depends on the applicability of these residues and being complex and different materials, it is necessary to characterize them. This study evaluates the waste of a fruit processing industry according to their basic physicochemical characterization. After sanitization, drying in a forced air circulation at 55 °C to constant weight, crushing in knives mill type Willye and packaged in airtight plastic containers at room temperature, the residues were subjected to physico-chemical analysis. The results demonstrated that the residues have very high carbohydrate content (56 - 76%) mostly consists of fibers, that may be used in the enrichment of whole foods, production of bioplastics, lignocellulosic biomass to second generation ethanol production or adsorptive processes. Except to acerola seed, that presents 5%, the moisture content was around 10%. The lipid content was important in the graviola seeds (25%), suggesting possible extraction of essential oils, usually present in seeds of fruits, with application in cosmetic/pharmaceutical industry. The protein content remained similar in waste, around 7%. The fixed mineral residue was around 3%, with the exception in banana peels (10%). The amount of pectin was quite significant in the passion fruit peel (27%), can be thought of in the extraction process, since this polymer has broad market in the food and pharmaceutical industry.

Keywords: waste, fruit pulp, peel, bioproduct.

CARACTERIZAÇÃO FÍSICO-QUÍMICA DE RESÍDUO DO PROCESSAMENTO DE POLPA DE FRUTAS: UMA ABORDAGEM SOBRE ALGUNS PROCESSOS DE RECUPERAÇÃO DOS BIOPRODUTOS

RESUMO

O Brasil é o terceiro maior produtor de frutas do mundo e, como resultado da necessidade de conservação e transformação, geram-se grandes quantidades de resíduos, tais como cascas, sementes e torta de filtro (bagaço) e a sustentabilidade do processo depende da aplicabilidade desses resíduos, os quais, por serem materiais complexos e diferentes, é necessário caracterizar. Este estudo avaliou os resíduos de uma indústria de processamento de frutas de acordo com sua caracterização físico-química básica. Após higienização, secagem em estufa com circulação forçada de ar a 55 ºC até peso constante, trituração em moinho de facas tipo Willye e embalagem em recipientes de plástico hermeticamente fechados, à temperatura ambiente, os resíduos foram submetidos à análise físico-química. Os resultados mostraram que os resíduos possuem um conteúdo de carboidratos muito elevado (56-76%), sendo principalmente constituído por fibras, que podem ser utilizadas no enriquecimento de alimentos integrais, produção de biopolímeros, biomassa lignocelulósica para produção de etanol de segunda geração ou processos de absorção. À exceção da semente acerola, que apresentou 5%, o teor de umidade dos resíduos esteve em torno de 10%. O teor de lípidios foi importante nas sementes graviola

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The demand for health and comfort has been a global trend, leading to fruit consumption increase. This development in trade in processed products in the country is due to factors such as shorter food preparation, full use and as close as possible sensory quality of food in nature (ABF, 2013).

Brazil is the third major fruit producer, after India and China, generating more than 40 million tonnes and making the country responsible for 6% of world production (Andrigueto et al., 2013). Of these, about 50% are processed by the beneficiary industries or small associations and cooperatives, that ensure the distribution of the fruits over long distances and during the off-season, as juices, pulps, jams, among others, generating a large amount of waste, normally used as animal feed (ABF, 2013). Depending on the fruit, the yield is around 50%, making food waste a matter for the treatment, prevention and minimization of environmental effects induced from their improper disposal (Galanakis et al., 2012).

Fruits like orange, banana, passion fruit and pineapple are very accepted worldwide, being Brazil a major producer of the small ones, where the first two are responsible for about 60% of the volume of fruits produced in the country. Exotic fruits like graviola, acerola, among others, have great acceptability in the internal market and the demand for its has been increasing (ABF, 2013).

The applicability of these residues may have a greater extent, as an additional protein enrichment and development of integral products, because they are rich in fiber, in addition to use in adsorption processes, lignocellulosic ethanol production and extraction of organic acids and enzymes. This work aims to characterize waste from the processing of orange pulp, passion fruit, graviola, pineapple and acerola and describe possible applications of these wastes in obtaining value-added bioproducts.

**INTRODUCTION**

**MATERIAL AND METHODS**

The waste, after juice/pulp extraction in the production unit (passion fruit, pineapple and graviola, at Coopeagro, in Maragogi-AL, and orange at Coopral, in Santana do Mundaú-AL) and banana, were collected and taken to the laboratory where it was applied sanitization with sodium hypochlorite at 100 ppm for 15 min. After, they were cut or separated into squares of 6 x 6 cm, approximately, on trays and placed in a forced air circulation oven at 55 ± 5 °C and 2 m.s⁻¹ air speed of until constant weight and, subsequently, ground on slicer type Willye, with 30 mesh sieve, and disposed in hermetic packaging plastics at room temperature.

The residues were classified into passion fruit peel, graviola seed, graviola waste (bark + seed + fiber pulp), graviola pulp, peel and pineapple pulp, banana peel, lime orange pulp and acerola seed.

Analyses were performed from moisture, ash, proteins, lipids, fiber and sugars. The moisture, fixed mineral residue (ash), lipids and crude protein followed by analytical standards of the Adolfo Lutz Institute (AL, 2005) and AOAC (2002). The moisture content was determined based on the product weight loss subjected to heating at 105 °C until constant weight. The ash, or fixed mineral residue, corresponds to the residue obtained by incineration at temperatures up to 550 °C until obtain a clear gray. The determination of total lipids was carried out directly in Soxhlet apparatus, with hexane as the extraction solvent. The crude protein determination was performed by the Kjeldahl method, using 6.25 as a factor for the protein nitrogen. The crude fiber content was determined by the method proposed by Hennemberg (1864) cited by Giger-Reverdin (1995), in which the sample undergoes digestion in acid medium followed by another in an alkaline medium, where the solid material remaining was quantified as crude fiber. The percentage of total carbohydrate was made by the above difference analysis. The pectin (Pe) quantification used the...
method proposed by Carvalho et al. (2002), in which the suitably ground sample is boiled in a slightly acidic solution, where it is solubilized in the amount of pectin present in the sample, followed by precipitation with calcium chloride, filtered, dried and weighed.

After chemical analyzes of lipids (L) protein (P), moisture content (M) fixed mineral residue (ash) (A) and fiber (F), the theoretical amount of carbohydrates (C) was obtained by difference, according to Equation 1.

\[
C = 100 - \left[ F + M + A + P + L \right] \quad (1)
\]

Analyses of variance and comparison of averages by Tukey test at 95% confidence level was conducted by ASSISTAT program, 7.6 Beta version (Silva, 2012).

**RESULTS AND DISCUSSION**

The residue drying lasted about 18 hours. By the physicochemical characterization (Table 1), it can be seen that the amount of carbohydrates present in biomass ranged between 56 and 76%, with the greatest results for the graviola waste, passion fruit peel, acerola seed, pineapple pulp and pineapple peel. This high amount of carbohydrates makes such residues interesting in various forms of reuse of their energy content and different processes, as substrate for solid state fermentation (SSF) aiming the enzymes production and protein enrichment, use as biosorbents, as additive in the production of functional foods, in the animal feed, in the syrups production or use as supplement energy in the fuel production, such as methane and second-generation ethanol.

Currently, the most common is to use them in animal feed, that leads to lower costs, since this step is responsible for 70% of animal production costs (Correia et al., 2006; Lousada Junior, 2006; Cross et al. 2013). Cruz et al. (2013), evaluating the by-products use from the cashew, orange, passion fruit and pineapple production in animal feed, show great ability of the residue to be used in complementing. Lousada Junior (2006) studied the completion of elephant grass with 5-20% of waste from processing pineapple, cherry, melon, guava and passion fruit from the nutritional characterization of the waste until the animal consumption. The completion advantage is that is a direct application, requiring no more processing steps. However, time between production and consumption influence the degradation of this waste, mainly due to water content, being a limiting factor to this practice.

### Table 1 – Physical-chemical characteristics of the waste.

<table>
<thead>
<tr>
<th>Waste</th>
<th>%F ±</th>
<th>%M ±</th>
<th>%A ±</th>
<th>%P ±</th>
<th>%L ±</th>
<th>%C ±</th>
<th>%Pe ±</th>
</tr>
</thead>
<tbody>
<tr>
<td>Graviola pulp</td>
<td>16.27</td>
<td>8.65</td>
<td>3.49</td>
<td>7.60</td>
<td>7.25</td>
<td>56.75</td>
<td>14.94</td>
</tr>
<tr>
<td>Graviola seed</td>
<td>8.27</td>
<td>4.12</td>
<td>4.05</td>
<td>10.35</td>
<td>23.86</td>
<td>49.35</td>
<td>9.83</td>
</tr>
<tr>
<td>Graviola waste</td>
<td>9.78</td>
<td>8.37</td>
<td>3.50</td>
<td>7.37</td>
<td>3.20</td>
<td>66.46</td>
<td>9.75</td>
</tr>
<tr>
<td>Passion fruit peel</td>
<td>13.79</td>
<td>8.82</td>
<td>2.02</td>
<td>4.68</td>
<td>0.99</td>
<td>69.70</td>
<td>27.88</td>
</tr>
<tr>
<td>Acerola seed</td>
<td>12.66</td>
<td>10.68</td>
<td>1.53</td>
<td>8.16</td>
<td>4.32</td>
<td>67.44</td>
<td>12.05</td>
</tr>
<tr>
<td>Pineapple pulp</td>
<td>10.08</td>
<td>8.68</td>
<td>3.62</td>
<td>4.02</td>
<td>1.26</td>
<td>72.34</td>
<td>5.43</td>
</tr>
<tr>
<td>Pineapple peel</td>
<td>5.52</td>
<td>9.38</td>
<td>2.99</td>
<td>4.84</td>
<td>0.84</td>
<td>76.00</td>
<td>2.12</td>
</tr>
<tr>
<td>Lime orange pulp</td>
<td>12.23</td>
<td>10.05</td>
<td>3.40</td>
<td>3.38</td>
<td>1.74</td>
<td>69.14</td>
<td>8.04</td>
</tr>
<tr>
<td>CV(%)</td>
<td>40.82</td>
<td>18.20</td>
<td>21.81</td>
<td>15.13</td>
<td>15.11</td>
<td>18.07</td>
<td>15.11</td>
</tr>
</tbody>
</table>

CV (%) is the coefficient of variation between the averages of the waste variables studied. The letters a, b, c, d, e are representative of the Tukey test, where numbers followed by letters or number of different letters differ statistically from each other at 5%.

Grouping the waste based on the analysis performed, it appears that passion fruit peel, possibly due to high pectin content, graviola seed, by the content of lipids, and pineapple peel, due to high concentrations of total carbohydrates, are the ones that excel.

The solid state fermentation (SSF) consists in the growth of microorganisms on a solid matrix, in which the water content is attached, without free water presence. The current research lines are directed to the protein enrichment of agro-industrial waste, detoxification of waste and/or production of aggregates compounds such as enzymes and metabolites (Rainbault, 1998).

The protein enrichment is suitable for waste owns unbalanced protein/carbohydrate content, when and they do not meet digestive requirements and ruminant fermentation, to optimize the growth of microbial flora and fiber digestion (Correia et al., 2006).
Araújo et al. (2011) enriched the mangaba flour seeds using Rhizopus oryzae, increasing up to 3 times the protein content of the residue in a 60% humidity and 40 °C fermentation temperature. Alexander et al. (2013) made a protein enrichment from pineapple waste processing (Ananas comosus) with the yeast Saccharomyces cerevisiae, which, after fermentation time, there was protein content about 3 times higher than the fresh waste. Santos et al. (2010), by the use of Aspergillus niger, evaluated protein enrichment waste from the processing of cupuacu, jackfruit, seriguela, pineapple and guava and, after 168 hours of cultivation at 35 °C, observed that 50% of these residues doubled their protein content.

As regards the production of enzymes by solid state fermentation (SSF), it must be verified slightly better the characteristics of the waste, because the microorganism will produce the enzyme of interest in larger quantities if the residue has the target compound in appreciable quantities, since it serves as an inductor. As an example, it mentions the passion fruit residue, which has a high pectin content (around 27% on a dry basis), heterogenous polymer that has applications in the juice industry, fermented, effluent, pharmaceuticals and fine chemicals, and can be used in the production of pectinolytic enzymes. Souza et al. (2010) studied the production of pectic enzymes using the passion fruit peel and the fungus Aspergillus niger CCT 0916, in a process with 40% humidity and 30 °C, with greatest activity (21 U.g⁻¹) achieved at 20 h of fermentation. Alcantara et al. (2007) evaluated the cashew stalk residue resulting from the extraction of the pulp for the pectinase production in Aspergillus niger, indicating that the waste moisture factor needs to be adjusted, but nothing has been discussed in relation to substrates and minerals. Sousa (2014) produced phenolic extracts from pineapple and guava waste, using the Rhizopus oligosporus fungi. Dantas & Aquino (2010) produced lipases from pumpkin seeds and avocado peel, with activities around 20-30 U.g⁻¹, producing crude extracts with activity between 2500 and 3500 U.g⁻¹.

The cell wall of plants owns, mostly, cellulose polymers, hemicellulose and lignin, besides pectins and proteins. Generally, the major part is cellulose, followed by hemicellulose and lignin. With the increase ethanol demand to values greater than 40 million liters per year, in this last decade, there is the need to look for new sources of fermentable sugar production, as the first generation substrates (sugarcane and corn, mainly) are no longer able to supply the whole demand. In this context, the lignocellulosic material enters like second raw material generation to obtain ethanol.

Table 2 shows that the tropical fruit industry wastes have high cellulose and hemicellulose contents, with several studies in order to use them to energy complementation, through the second-generation ethanol production. There are two main strands to the use of this waste: (1) centered on breaking the structure of fermentable sugars; (2) use for the production of enzyme complexes by solid state fermentation, especially cellulases.

<table>
<thead>
<tr>
<th>Waste</th>
<th>% Carbohydrates</th>
<th>%Cellulose</th>
<th>%Hemicellulose</th>
<th>%Lignin</th>
<th>Reference</th>
</tr>
</thead>
<tbody>
<tr>
<td>Graviola waste</td>
<td>-</td>
<td>13.30</td>
<td>21.00</td>
<td>12.90</td>
<td>Pereira et al. (2008)</td>
</tr>
<tr>
<td>Pasion fruit peel</td>
<td>12.98</td>
<td>40.35</td>
<td>25.69</td>
<td></td>
<td>Rogerio et al. (2011)</td>
</tr>
<tr>
<td>Acerola waste</td>
<td>83.61</td>
<td>35.07</td>
<td>17.17</td>
<td>20.11</td>
<td>Lousada Junior et al. (2006)</td>
</tr>
<tr>
<td>Pineapple peel</td>
<td>82.00</td>
<td>34.47</td>
<td>27.74</td>
<td>14.33</td>
<td>Ferreira et al. (2009)</td>
</tr>
<tr>
<td>Lime orange pulp</td>
<td>16.19</td>
<td>27.85</td>
<td>8.79</td>
<td></td>
<td>Retore et al. (2010)</td>
</tr>
<tr>
<td>Banana peel</td>
<td>18.00</td>
<td>-</td>
<td>17.00</td>
<td></td>
<td>Souza et al. (2012)</td>
</tr>
</tbody>
</table>

For the lignocellulosic enzymes production by FSS, the waste from wood processing, corn, sugarcane, wheat, rice and cassava are the most used, with fruit waste little explored (Couto & Sanromán, 2005 Martins et al., 2011). Krishna (1999) studied the production of cellulolytic enzymes using *Bacillus subtilis* and observed that banana peel would be able to produce commercially this class of enzymes. Reddy et al. (2003) also used banana peel to produce cellulases, hemicellulases and ligninases, using two species of microorganism *Pleurotus gender*. Ferreira et al. (2011) produced endoglucanases (type of cellulase) from cajá processing waste and obtained high activities, maximizing at 50% humidity and 72 hours of fermentation (531 U.mg⁻¹).

The pineapple peels have high hemicellulose content (27.74%), highly heterogeneous polymer that requires a large complex of enzymes. It is suggested their use in the production of hemicellulases, since it is a raw material with all heterogeneous components from biomass, which may be an advantage to improve the composition of the enzymatic complex.

Xylan is the main hemicellulose oligosaccharide, it has been many efforts directed to surveying hemicellulases complete degradation of xylan. Require two enzymes: (1) endo-1,4-β-xylanase, which attacks xylan main chains; and (2) β-xylosidase, which hydrolyze the xylo-oligosaccharides to xylose. It is required other enzymes, said accessory, since the xylan is the major oligosaccharide, but there are others in the hemicellulose chain. As well as cellulases, the metagenomic can be used in its screening (Machado, 2013).

Souza et al. (2012) evaluated the banana peel as substrate for second generation ethanol production, and found that the yield of fermentation was similar to sugarcane. Grohmann et al. (1995) studied the acid and hydrothermal pre-treatment with citric residue, and Macedo et al. (2011) using pequi peels, reached reasonable levels of ethanol.

In view of the results, Figure 1 illustrates the main alternative using waste processing studied fruit.

![Figure 1: Prospects for the waste use from the fruit processing.](image)

Another alternative is to use them dried, after washing, in adsorption processes, due to the presence of many fibers. Ströher et al. (2012) used orange bagasse to remove dye jeans in washing water and achieved a reduction of about 50% in the color and turbidity of the effluent. Boniolo et al. (2010) used banana peels to remove uranium ion from aqueous solution and Feng et al. (2011) studied the cadmium removal, nickel and lead, detecting high affinity for biomass to orange peel.

The seeds, due to the high content of lipids and essential oils, can be an extraction source. Essential oils of orange seeds, passion fruit, tomato, guava, pink lemon and lemon were studied by Reda et al. (2005) and George & Kobori (2005). Nunes et al. (2009) mentioning the importance of using waste from the citrus industry, having an important activity in the extraction of essential oils from their...
seeds, which have triglycerides, terpenes, alcohols, esters and aldehydes.

The adequacy of these waste to a particular process not only recovers the deposited energy in them, as it becomes more sustainable production chain and directs the management of solid waste.

CONCLUSIONS

Brazil has a natural tendency to the production of various types of fruit, such as tropical and exotic fruits, thanks to the climate and agricultural nature, being the third largest producer. The processing of these fruits generates huge amounts of waste, about 50%, besides the post-harvest losses. Among the characterized waste (graviola, acerola, passion fruit, orange, pineapple and banana), it was noticed a large amount of total carbohydrates, between 56 and 76%, especially in the peel and graviola waste, orange, pineapple, passion fruit and acerola seeds. The passion fruit peel has a high pectin content (27%) and graviola seed, lipids (24%), being most appreciated for producing enzymes by solid state fermentation, extraction of pectin and oil extraction, respectively. Many studies try to make possible the by-products recovery from them, among which detach the animal feed, the second-generation ethanol, biosorption, the enzymes production and protein enrichment by solid state fermentation and extraction of essential oils from seeds. This will certainly contribute to better management of solid waste generated.

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