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Improvement of bixin extraction from annatto seeds using a screen-topped spouted bed

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ABSTRACT

Bixin is the most used carotenoid in the food industry. It has been conventionally extracted from annatto seeds (*Bixa orellana* L.) using alkaline solutions or organic solvents. These methods, in addition to require a subsequent unit operation of bixin separation, can generate toxic waste. Thus, the demand for more environmentally attractive solutions leads to the study of alternative extraction technologies, such as mechanical extraction. This study aimed to investigate the mechanical extraction of bixin from annatto seeds using a screen-topped spouted bed unit. An experimental study was performed to verify the influence of process variables – such as mass of seeds, height of the screen inside the equipment, and air flow rate – on product yield and purity. Regression techniques were used to quantify the effects of these variables. The optimum conditions for the mechanical extraction were determined using a differential evolution algorithm. From the experimental work, it was extracted an annatto powder with variable contents of bixin, but always commercially attractive (over 30% of bixin). Using the optimization process, the conditions that led to the best extraction efficiency (for powder and bixin) and greater purity powder (above 65%) were identified. The experimental values closely agreed with the predicted values.

1. Introduction

Annatto is a natural dye obtained from the pericarp of the seeds of *Bixa orellana* L. and its extracts, widely used in food, are finding increasing interest also for their application in the pharmaceutical and cosmetics industry [1,2]. This colorant imparts colors ranging from yellow to red due to the concentration of color compounds in the solution [3]. Besides, compared to other natural dyes, annatto extracts are relatively inexpensive, thus increasing their commercial relevance [4,5].

The main pigments extracted from annatto seeds are the carotenoids cis-bixin and cis-norbixin (Fig. 1). The first is a fat-soluble compound and is a major component in the extracts obtained when preparing oily suspensions or organic solvents. Cis-norbixin is a water-soluble compound and is thus found mainly in the form of alkaline solution extracts [2].

Three commercial processes have been commonly applied to extract the pigment from dehydrated annatto seeds: direct extraction into oil, indirect extraction with solvents, or direct extraction into aqueous alkali solutions [6]. In the first method, the abrasion of the exocarp submerged in heated vegetable oil (70 °C) produces concentrated suspensions of pigment, but they may contain degradation products, because the annatto pigment can degrade and form several products at high temperatures [6,7]. Industrially, the most used process is the extraction into alkaline solution, usually potassium or sodium hydroxide [8]. This process is known to transform bixin (present in the seeds as an ester) into norbixate, a water-soluble diacid salt [9]. The removal of the alkaline solution from the bixin-rich extract and the seeds requires several subsequent unit operations, whose energy costs are elevated. Many solvents, such as acetone, chloroform, ethanol, ethyl acetate, and hexane are used in the extraction of annatto pigment due to its strong solubility [10–12].

Various techniques have been studied to develop clean, low environmental impact technologies to extract bixin from annatto seeds: the supercritical technology [13–16], the extraction from defatted seeds [16–19], the microwave and ultrasound-assisted extraction [18,20,21], and the enhancement of the conventional extraction method using water or ethanol as solvent [16,18,21,22].

The mechanical attrition of annatto seeds is an alternative physical method which wears the pigment layer by friction. This method presents some advantages, such as shortening the processing time, reducing or eliminating solvent consumption, increasing the extraction yield, and improving the quality of the extracts [23]. This technology uses a simple apparatus, such as a ball mill or a

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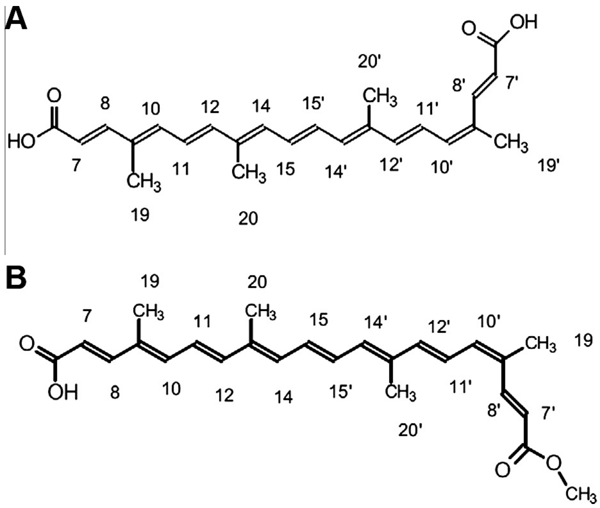

Fig. 1. Chemical structure of apocarotenoids cis-bixin (A) and cis-norbixin (B) [2].

spouted bed, so that they are suitable for installation in annatto plantations, adding value to the final product. The major advantage of using a spouted bed is to have a better control of the process in comparison with other devices [24–26].

The spouted bed technique is an alternative to fluidization of particulate solids too coarse for good fluidization [27]. Spouted beds are gas-particle contactors; the gas is introduced into the spouted bed through a single nozzle at the center of a conical or flat base, partially filled with particles [28]. The gas–solids flow in a spouted bed can be divided into three regions: a spout at the center, where gas and particles rise at high velocities; a fountain zone, where particles rise to their highest positions and then rain back onto the surface of the annulus; and an annulus zone between the spout and the column wall, where particles move downward as a dense phase, with counter-current percolation of the fluid [29]. This technique has been applied to many industrial processes, such as drying of granular materials, blending of polymer chips, coating, and granulation of fertilizers and other materials [30–33].

Besides handling coarse particles, spouted beds have an effective fluid–solid contact in their structural and cyclic flow patterns [34], which offers an excellent alternative to mechanically extract bixin from the surface of annatto seeds. The fluid dynamics characteristics of the spouted bed allow the combination of impact and seed drying in the same unit [35] and generate a good quality final product.

Some works have been developed with the aim to obtain bixin from annatto seeds using spouted beds. Passos et al. [36] studied the powder production rate in a conical spouted bed unit. Cunha et al. [23] concluded that the draft tube was the variable that most affected powder extraction. Barrozo et al. [25] quantified the fluid dynamic aspects, the mass extracted, the yield, and the bixin content. These previous studies have highlighted that the gas flow rate and the presence of the draft tube are the operating parameters that strongly influence bixin recovery due to the high particle attrition rates they promote. The gas canalization in the draft tube in the central part of the bed accelerates the particles, and travel up the draft tube under the influence of the competing forces of fluid drag, particle-to-wall friction, and momentum interchange between particles. With the aim to increase bixin recovery from annatto seeds, the present research proposed the use of a new configuration of the spouted bed with a draft tube: the screen-topped spouted bed. The influence of the position of a stainless steel screen along the cylindrical part of the spouted bed was investigated, and the mass of seeds and the gas flow rate were also studied. The best results for these operating parameters were determined by means of an experimental design, multiple regression analysis, and process optimization using genetic algorithms.

2. Materials and methods

2.1. Materials

Annatto seeds were obtained from the city of Rio Vermelho (Minas Gerais State, Brazil) and stored at −18 °C. They were protected from light by keeping in dark polyethylene bags. The solvents used in the analysis were chloroform, hexane, and acetone; all of them were analytical-grade reagents provided by Synth®.

2.2. Seeds characterization and analysis

A batch of seeds had its characteristics analyzed: moisture, protein, ash, total lipids and bixin content. The moisture, protein and ash contents were determined according to the official methods by the Association of Official Analytical Chemists [37]. The seed oil content was quantified by gravimetric analysis, in which evaporation was performed until drying the total volume of the sample in a Soxhlet apparatus with hexane. The carbohydrate content was determined by difference. All experiments were carried out in duplicate.

The pigment content of the seeds was determined by an organic extraction method, which consisted in using chloroform to extract pigment until it was totally removed from seeds. This extraction was conducted at 50 °C using an equilibrium cell coupled to a reflux condenser, whose purpose was to prevent the loss of desirable compounds (volatiles). The temperature of the bath connected to the Soxhlet was 5 °C. These conditions were based on the research conducted by Silva et al. [38], whose results showed the good performance of chloroform as a solvent combined with temperatures lower than 80 °C, which cause minimum degradation to bixin. The bixin content of the seeds was determined using the spectrophotometric method according to the FAO/WHO Joint Expert Committee on Food Additives [39]. The sample absorbance was measured in a 1-cm quartz cuvette at 487 nm using a UV–visible spectrophotometer. The percentage of bixin in the annatto seeds was calculated, then, according to Lamber–Beer law, using $E^{1\%}_{1\text{cm}} = 3090$ [39], in Eq. (1):

$$\text{Bixin } (\%) = \frac{A \cdot V_1 \cdot \ldots \cdot V_n}{E \cdot m_{\text{sample}} \cdot V'_1 \cdot \ldots \cdot V'_n} \tag{1}$$

In which:

A = average absorbance of the samples;
V_i = dilution volume (i = 1, 2, ..., n);
E = absorptivity coefficient E (equal to 3090 for Bixin);
M_{sample} = sample mass, in grams;
V'_i = volume of aliquot for dilution (i = 1, 2, ..., n).

2.3. Spouted bed extraction

The spouted bed experiments were conducted in an experimental apparatus. Fig. 2 shows a schematic diagram of the equipment.

The experimental set-up was composed of a conical–cylindrical spouted bed (1), a 7.5-hp air compressor (2), an orifice flow meter (4), and a pressure transducer (3). The concentrated bixin powder extracted was collected in a receptacle (7) protected from light, which was connected to the underflow of a 10-cm-diameter, Stairmand cyclone (8). The pressure transducer signals were transmitted to a computer via a data acquisition system (6) and processed by the software LabVIEW™ 7.1. The spouted bed (1) consisted of a stainless steel cylinder with a conical bottom.