



The Effect of Low Pressure Oven Drying of Chicory Root on Quality and Extraction Time of Inulin

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ABSTRACT

Chicory (*cichorium intybus*) is a perennial herbaceous plant of Asteraceae family, and resistant to cold climate and frost. Chicory root containing 70-94% inulin, based on dry weight is considered as a major source of inulin for industrial production. Inulin is a fructologosaccharide and is a substitute for fat and sugar that is not digestible, but in the colon, it is used by bifidobacteria. Some of these bacteria are used as a periosteum and are very useful for human health. Drying is one of the most important post-harvesting processes of medicinal plants, which plays a major role in the quantity and quality of its active ingredients. To prevent fresh chicory root from deterioration, it should be dried for long-term storage. The aim of this study was to investigate the effect of drying in low pressure on the quality and time of extraction of inulin from Chicory root. For this purpose, the chicory roots were first dried at 80 °C under two different oven pressure of 35000 and 75000 Pa for one hour, then the inulin was extracted using diffusion process in hot water for 70 and 150 min. For qualitative evaluation, extracted inulin was analyzed through High Performance Liquid Chromatography (HPLC) with refractive index (RI) detector. Based on results from HPLC analysis, the dried sample at 750 mbar and extraction time of 150 min had the highest amount of inulin, while the dried sample at 750 mbar and the extraction time of 70 min had the lowest amount of inulin.

INTRODUCTION

Chicory with the scientific name (*Cichorium intybus*) is a perennial herbaceous plant from the Asterales family with purple-blue flowers at a height of 0.5 to 1.5 m and resistant to cold and frost. Today's uses of chicory are more focused on the root and its active ingredient, i.e. inulin. Chicory root contains 70 to 94% inulin based on dry weight, which is considered as the most important root source for the industrial production of inulin due to the production of inulin with a long and stable chain of fructans. The amount of chicory root production based on dry weight is reported to be around 11 to 16 tons per hectare, which leads to the production of 8 to 12 tons per hectare of inulin; Of course, if there are no losses during the extraction and purification process (Smits and Leenheer, 2003). Inulin is naturally produced in more than 36,000 types of plants belonging to 10 families and 1,200 species, and after starch and cellulose, it is the most abundant carbohydrate naturally present in plants. Inulin, the main storage polymer it is a member of the composite family, such as chicory, dandelion, artichoke, garlic, onion, banana and sedge. Today, inulin has become a useful raw material in the food industry, which provides the possibility of improving the nutritional properties

of various products, replacing fat and sugar as a low-calorie food, and controlling body weight in order to reduce energy intake. and blood sugar reduction is used (Kosari, 1993).

Drying, which is one of the important steps after harvesting medicinal plants and is considered one of the most widely used methods of preserving agricultural products, plays an important role in the quantity and quality of their effective medicinal substances. The use of inappropriate drying methods leads to the loss of plant organs or the effective substances contained in them (Moses *et al.*, 2014; Methakhup *et al.*, 2005).

Chicory plant must be harvested within a short period of time. To avoid the product deterioration, it must be processed immediately. Since the processing capacity of the industry is not high enough to handle the high volume of crop yield, an appropriate technique of drying is needed to preserve the product for later processing. Careful consideration must be given to drying technique chosen in such a way that the Inulin extraction efficiency from its roots during later stages of processing remains unaffected and high quality Inulin be obtained (Vakili and Hojjat al-Islami, 2013).

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MATERIALS AND METHODS

Preparation, storage and preparation of samples

Chicory roots grown under natural field conditions were randomly sampled from the suburbs of Sarab city, East Azarbaijan province. After harvesting, the samples were placed in double-walled plastic bags and kept in a refrigerator at 4 °C until the experiments were performed. Before doing the drying tests, chicory root samples were taken out of the refrigerator and kept at room temperature (20 °C) for 12 h to reach temperature equilibrium with the environment (Tavakoli Kardler *et al.*, 2016).

Determining the initial moisture content of the samples

To determine the initial moisture content of chicory roots, it was done according to the AOAC standard (AOAC, 1995). From chicory root, 5 samples with specific weight were selected and placed in an oven at 100 °C for 24 h (Toneli *et al.*, 2008). According to Equation 1, the initial moisture content of the samples was 79.25% w.b.

The measurement of the moisture content of plant material is calculated based on wet weight or dry weight according to the following equations (Juliano, 1984).

$$M_{w.b} = \left(\frac{W_w}{W_w - W_{dm}} \right) \times 100 \quad (1)$$

Where, W_w is initial weight of plant material before drying, W_{dm} is weight of plant material after 24 h of drying at 100 °C and W_{wb} : initial moisture content of plant material based on wet weight (Noh *et al.*, 2015).

Drying method

To dry the samples from the vacuum oven (BINDER, VD 23, Germany) (Fig. 1) used. Its vacuum pump had a one-horsepower, single-phase 230-volt electric motor with a frequency of 50 to 60 Hz. Before starting the experiment, the temperature of the oven reached a stable state, the oven was kept at the desired temperature for one hour.



Fig 1. Schematic diagram of pendulum arm position before and after stalk cutting

The leaves and herbaceous parts of the samples were separated from the roots after initial cleaning. The roots were washed well with water and chopped with a sharp knife into almost uniform pieces of 3 x 6 x 45 mm in size (Vakili and Hojjat al-Islami, 2013).

The experiments were carried out under a temperature of 80 °C and a pressure of 350 and 750 mbar during the drying process for 60 min. For drying in each experiment, three samples of the prepared

slices were selected and placed in a vacuum oven at the same time, and the drying process was performed on them. After drying for 60 min, the dried slices were removed from the dryer and their weight loss procedure was measured and recorded using a digital scale with an accuracy of 0.001 grams (Model PTY A200, Made in Shinko Japan). Then, dried root slices were transferred into double-walled plastic bags and kept in a refrigerator at 4 °C for inulin extraction.

Inulin extraction

Extraction of inulin from chicory root was done using hot water diffusion method (Vakili and Hojjat al-Islami, 2013). In this way, the dried samples were placed in a beaker and distilled water at 60 °C was added to them with a ratio of 1 to 5 according to the weight of the wet sample. Then, using 0.1 normal hydrochloric acid, the pH of the sample was brought to 5. PH was measured with a pH METER, (Model pHs-25C, China). Then the samples were placed in a bain-marie bath with a temperature of 60 °C and 2 cc of the extract was sampled at intervals of 70 min and 150 min. After completing the sampling process, all the samples were filtered with the help of a 0.45micron needle filter and poured into separate falcons and kept at a temperature of 4 °C under standard conditions to evaluate the quality of inulin extracted by chromatography. High performance liquid chromatography (HPLC) is used.

Quantitative measurement of inulin using HPLC device

Isolation, identification and determination of the small amount of inulin studied in this research using a high-performance liquid chromatography device, 1100 series model, manufactured by the American company Agilent, equipped with an injection loop with a volume of 20 microliters, four gradient pumps Solvent, degassing system, column oven (set at 45°C) and refractive index (RI) detector were used. Separation was performed on an octadecylsilane column (25 cm long, 4.6 mm internal diameter and 5 µm particle size ZORBAX Eclipse XDB) manufactured by Dr. Mainsch, Germany. Chemstation software was used for data processing. In order to separate inulin better, Elution program was used, for this purpose; the mobile phase of 100% water with a flow of 1 ml/min was used. The separation time was 10 min. In addition, standard inulin powder from Aldrich Company, made in Germany, with a sincerity of 99.5% was used.

Determination of inulin

The external standardization method is used to determine the extracted inulin in the samples of different dried chicory roots. To perform this method, first a sample was prepared using standard (pure) inulin and analyzed by HPLC. The result of the analysis, which is a refractive index in terms of time, was recorded. This chart has peaks at different times. These times are called its inhibition time. The inhibition time is the same for each material using a fixed device (in terms of column specifications). In other words, each combination has its own inhibition time. Therefore, it is possible to identify different compounds in the sample based on the inhibition time.

Also, the size of this peak expresses the concentration of the desired substance. In this way, the area under the graph in the peak part will be according to the concentration of that compound.

In this article, to determine the inhibition time of inulin, a standard sample with a concentration of 1% of pure inulin was injected into the column using the mentioned HPLC device. Distilled water was used to prepare the standard sample. Based on the inhibition time of inulin, which was determined using a standard sample, the inulin extracted from different chicory root samples was identified. Since the amount of inulin concentration in the standard

sample was known, based on the area under the peak of inulin and comparing it with the standard sample, the concentration of inulin extracted from different chicory root samples was calculated.

RESULTS AND DISCUSION

Standard inulin results

As mentioned in the materials and methods section, a standard sample of pure inulin was prepared to determine the concentration of inulin extracted from chicory root. The HPLC results for this standard sample are shown in Fig. 2. As can be seen, the inhibition time for inulin is 2.95 min and the area under this part of the curve, which is the peak related to inulin, is proportional to the concentration of inulin and is equal to (nRIU*s) 89422.24 is since the concentration of inulin in this sample is known and equal to one percent, the concentration of inulin in other samples can be determined according to this number.

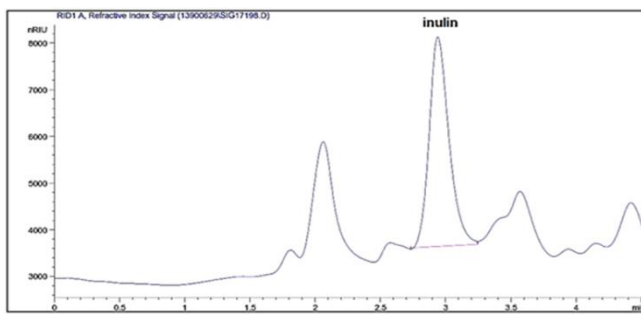


Fig 2. HPLC analysis result of pure inulin

The results of the research conducted to measure the amount of inulin in multivitamins indicated the success of the HPLC method to determine the amount of inulin along with the RI type detector. Their results showed that the inhibition time for standard inulin is equal to 6.89 min (Retnaningtyas, 2012). Also, in another research to measure the amount of inulin in Fructo-oligosaccharide (a type of probiotic that is a beneficial bacterium for the digestive system) was performed and the inhibition time of inulin was obtained at 5.85 min (Petkova *et al.*, 2014). This difference between the values of inhibition time is caused by the difference between the size and working temperature of the column of the HPLC device used.

The results related to the dried sample with a pressure of 750 mbar and an extraction time of 70 min

The results of HPLC analysis for inulin extracted from chicory root with a drying pressure of 750 mbar, a drying temperature of 80 °C and an extraction time of 70 min are shown in Fig. 3. As described, the inhibition time of inulin was obtained as 2.95 min, therefore the peak in this graph at 2.959 min is related to inulin and the concentration of extracted inulin is proportional to the level below the corresponding peak.

In this sample, as indicated on the graph, the area under the curve in the peak area related to inulin is equal to (nRIU*s) 438169. According to the area under the curve for pure inulin with a specific concentration, the concentration of inulin in this sample is 4.9%.

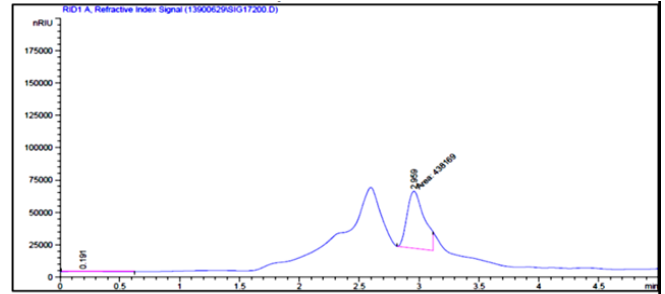


Fig 3. HPLC results for dried sample with 750 mbar pressure and 70 min extraction time

The results related to the dried sample with a pressure of 350 mbar and an extraction time of 70 min

The HPLC results of inulin extracted in 70 min from dried chicory root at a pressure of 350 mbar and a drying temperature of 80 °C are shown in Fig. 4. Based on the specified inhibition time for inulin, based on the standard sample, the peak corresponding to inulin was determined and to obtain the concentration of inulin in this sample, the area under the corresponding peak used and compared with the standard sample of inulin.

In this sample, as indicated on the graph, the area under the curve in the peak area related to inulin is equal to (nRIU*s) 458619. According to the area under the curve for pure inulin and its specific concentration, the concentration of inulin in this sample is equal to 5.21%.

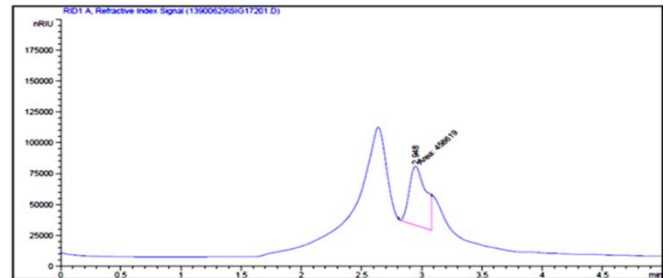


Fig 4. HPLC results for dried sample with 350 mbar pressure and 70 min extraction time

The results related to the dried sample with a pressure of 750 mbar and an extraction time of 150 min

Dried chicory root has been extracted for inulin with a pressure of 750 mbar and a temperature of 80 °C. The extraction was done in 150 min. The results of HPLC analysis related to this sample are shown in Fig. 5. The peak related to inulin is the same as the peak at the time of 2/956. As can be seen, the area under the peak related to inulin is equal to (nRIU*s) 612763. Therefore, the concentration of inulin in the present sample is equal to 7.57%.

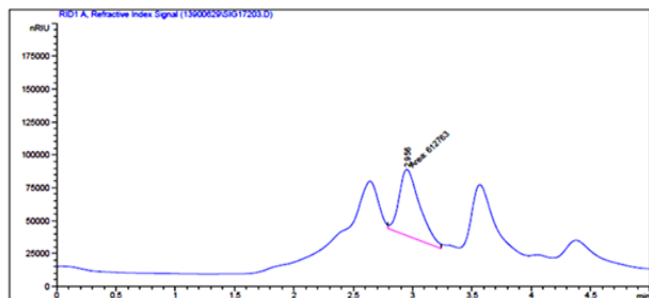


Fig 5. HPLC results for dried sample with 750 millibar pressure and 150 min extraction time

The results related to the dried sample with a pressure of 350 mbar and an extraction time of 150 min

The results of the inulin sample extracted in 150 min from dried chicory root with a pressure of 350 mbar and a temperature of 80 °C are shown in Fig. 6. According to what was said, the peak time of 2.923 min is related to the inulin present in this sample. Therefore, according to the area under the curve in this part (peak) which is equal to (nRIU*s) 490043 and according to the area under the curve of the peak related to inulin in the standard sample, the concentration of inulin in this sample is equal to 5.69.

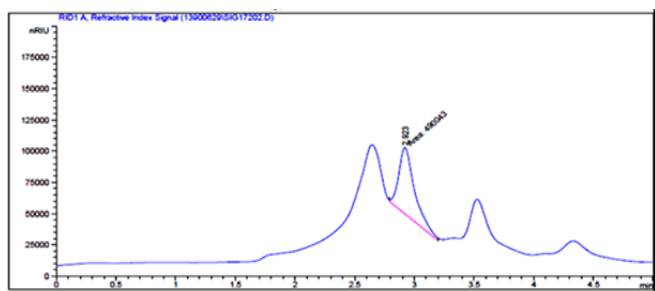


Fig 6. HPLC results for dried sample with 350 millibar pressure and 150 min extraction time

CONCLUSION

Based on the results obtained from this research to investigate the effect of drying pressure and inulin extraction time on the concentration of inulin extracted from chicory root, the general conclusion is presented as follows.

- The highest concentration of extracted inulin corresponds to the dried sample with a pressure of 750 mbar and an extraction time of 150 min.
- The lowest value of extractable inulin concentration corresponds to the dried sample with a pressure of 750 mbars and an extraction time of 70 min.
- There is no significant difference between the concentration of inulin in samples dried with 350 millibar pressure and extraction times of 70 and 150 min.
- It can be concluded that the drying pressure did not have a great effect on the concentration of extracted inulin.
- The concentration of extracted inulin increased with increasing extraction time.

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