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Inulin Content in Chipped and Whole Roots of Cardoon after Six Months Storage under Natural Conditions

Luigi Pari [1], Vincenzo Alfano [1,*], Walter Stefanoni [1], Francesco Latterini [1], Federico Liuzzi [2], Isabella De Bari [2], Vito Valerio [2] and Anna Ciancolini [3]

[1] Consiglio per la Ricerca in Agricoltura e l'Analisi dell'Economia Agraria (CREA)—Centro di Ricerca Ingegneria e Trasformazioni Agroalimentari, Via della Pascolare, 16, 00015 Monterotondo, Italy; luigi.pari@crea.gov.it (L.P.); walter.stefanoni@crea.gov.it (W.S.); francesco.latterini@crea.gov.it (F.L.)
[2] ENEA Centro Ricerche Trisaia, 75026 Rotondella, Italy; federico.liuzzi@enea.it (F.L.); isabella.debari@enea.it (I.D.B.); vito.valerio@enea.it (V.V.)
[3] NOVAMONT SpA, 28100 Novara, Italy; anna.ciancolini@novamont.com
* Correspondence: vincenzo.alfano@crea.gov.it; Tel.: +39-06-9067-5315

Abstract: Industries currently rely on chicory and Jerusalem artichoke for inulin extraction but also cardoon is proved to synthetize and store high quantity of inulin in roots as well. Cardoon is a multipurpose crop, well adapted to marginal lands, whose main residues at the end of cropping cycle consist of roots. However, cardoon roots are a suitable source of inulin, that is of high interest for new generation biodegradable bioplastics production. On the other hand, a sustainable supply chain for inulin production from cardoon roots has not been developed yet. In particular, in the inulin supply chain the most critical part is storage, which can negatively affect both cost and inulin quantity. In the present study the effect on inulin content in cardoon roots stored as dried chipped roots (CRt) and dried whole roots (WRt) was investigated in a 6-month storage trial. Our findings suggest that chipping before storage did not affect the inulin content during the storage. Furthermore, it reduced the time needed for drying by 33.3% and increased the bulk density by 154.9% with the consequent reduction of direct cost for drying, transportation and storage.

Keywords: Cynara roots; biorefinery; marginal lands; multipurpose crop; fermentable sugars; agricultural residues exploitation

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1. Introduction

Cardoon (*Cynara cardunculus* L.) is one of the most promising feedstocks for biorefinery in the Mediterranean areas since it is a multipurpose perennial crop well adapted to drought environments and low productive marginal lands [1–4]. The cultivation of this species mainly focuses on the exploitation of the aerial biomass as seeds, leaves and stalks [5–7]. The vegetable oil extracted from seeds is rich in monounsaturated fatty acids useful to produce important intermediates such as azelaic acid or pelargonic acid, that are highly demanded by synthetic fertilizer industries as well as cosmetic industries worldwide [8,9]. On the other hand, leaves and stalks represent an important source of lignocellulosic biomass potentially suitable for the production of intermediate compounds, like bioethanol and Bio-butanediol, which are widely used for producing bioplastics [10–12].

However, the potential of cardoon as a multipurpose crop has not been fully exploited yet, in particular regarding the presence in the roots of inulin suitable for nutraceutical, pharmaceutical and other biorefining applications [13–15].

Inulin is a linear fructan, i.e., a polymer of fructose units linked by β (2 → 1) glycosidic bonds with a variable degree of polymerization (DP), between 3 and 60, and usually a glucose molecule at the end [16]. Inulin can be used in food and pharmaceutical industry for several purposes, such as prebiotics to stimulate the growth of probiotic gut bacteria, for nutritional purposes as low caloric soluble dietary fiber and also as a mediate sugar

and lipid metabolism in diabetic and hypercholesterolemia [17]. In medicine, inulin is also used as a diagnostic agent for the determination of kidney function [18]. In the biorefinery industry, inulin and inulin-rich biomass are gaining interest for the production of fructose by enzymatic hydrolysis of inulin, as alternative way to the current approaches based on acid hydrolysis of sucrose [19,20]. In the biorefinery, moreover, the availability of fermentable sugars is crucial to produce ethanol, and inulin is a good feedstock for bioethanol production by fermentation after hydrolysis [21,22].

Inulin is a reserve carbohydrate accumulated mainly in the roots and tubers of many plants belonging to the Asteraceae family, like Cardoon [16].

Among the Asteraceae family plants, Chicory (*Cichorium intybus* L.) and Jerusalem artichoke (*Helianthus tuberosus* L.) are currently the major industrial sources of inulin [23]. In a comparative study aimed at evaluating different types of inulin, extracted from Cardoon roots, Jerusalem artichoke tubers and Chicory roots, the inulin amount resulted respectively in 115, 390 and 550 g kg^{-1} of d.m. [24].

In the perspective of new generation biorefineries and the circular bioeconomy concept, the recovery of inulin from cardoon roots at the end of crop cycle, in addition to various high added-value raw materials from seeds and stalks, seems to be an interesting opportunity. However, the full development of an effective value chain for the biochemical industries, implies well-organized logistics. Storage phase, in particular, has a high impact on the quality of the raw material and on the overall costs of the value chain [25].

Effects of storage conditions on inulin content have been investigated in different inulin-containing crops. In the case of the storage of Jerusalem artichoke tubers, inulin composition remained stable under frozen storage (−18 °C) during 3 months of study, while a significant degradation of inulin to sucrose and fructo-oligosaccharides was observed after 4 weeks when the storage was performed at 4 °C [26]. In another study, inulin hydrolase activity in the tubers of Jerusalem artichoke peaked at the 15th day of storage at ambient conditions: inulin underwent depolymerization causing a decrease in inulin content and an increase in soluble sugars [27]. In a 28 day storage trial a decrease of 70% and 96% was reported for artichoke heads after storage at 4 and 18 °C, respectively. Similarly, in storage of sliced artichoke heads at 4 °C, about 60% decrease of inulin content was observed during the first 11 days of storage [28].

In addition to the temperature, moisture plays a key role to start the enzymatic hydrolysis of the inulin. For example, it has been observed that the enzymatic hydrolysis of the inulin during storage of chicory roots depends on the moisture content, while the generated sugars favored the loss of material as a result of cell respiration and microbial activity [29]. In fact, microorganisms require minimum thresholds of moisture to maintain and optimize metabolism, that is the breakdown and the consumption of the sugar-based components of dry matter. Moisture below 10% is generally considered to be low enough to prevent microbial degradation and allow for safe long-term storage of biomass [30]. In this framework, the thermal drying technologies have gained interest as effective tools for extending the length of storage as well as reducing the handling cost and ease the transportation that affect the value chain at the industrial level [31,32].

On the other hand, a suitable storage system for a biorefinery has not only to focus on the capacity of keeping a high content of a given product, but there is also the need of finding a suitable solution regarding the economic sustainability, with particular reference to transport costs which can have a substantial impact on the overall value chain [33,34]. Under this point of view biomass chipping is an interesting approach. Indeed, the higher bulk density achieved by the chipped material improves the logistics by reducing the space needed during transport as well as in the storage area [35]. On the other hand, chipping increases the exposed surface area and reduces the air permeability [36] with an expectable opposite effect on drying efficiency as well as on the maintenance of dry matter and inulin content.

In order to set up a suitable value chain for inulin production from cardoon roots biomass, there is the need of investigating a storage system which combines effective long-

term maintenance of inulin and economic sustainability. Considering the current lack of knowledge on this particular topic, a specific task of the Italian Project Cometa-Autoctone Mediterranean crops and their valorization with advanced green chemistry technologies (funded by Ministry of Education, Universities and Research), was addressed to develop an effective handling and storage strategy for cardoon roots aimed to inulin production. In this framework the present study aimed to investigate the effect of chipping and drying approach on inulin content of cardoon roots biomass over 6 months of storage.

2. Materials and Methods

2.1. Location of the Field and Plant Material

Cardoon roots were taken in May 2020 from three year old plants cultivated in Terni (42.561335 N latitude, 12.62860 E longitude) (Umbria Region, Central Italy) on clay soil. Cardoon (cultivar Trinaseed) were grown from seeds sowed in November 2017 with a precision sowing machine at the rate of 3.0 kg/ha of seed and spacing distance of 0.75×0.17 m.

At the sowing, the seedbed was prepared between October and November with ploughing at 20 cm, followed by harrowing at 10–15 cm. During the first year, mineral fertilization with 46 kg of P2O5 and 64 kg/ha of N was added, and a.i. pendimethalin was used to control weeds. Starting from the second year, 46 kg/ha of P2O5 and 18 kg/ha of N were applied during the vegetative growth of the plants in autumn and in the early spring before the stem elongation phase. The fertilization rates were calculated on the basis of the soil fertility and crop nutrient uptake. Crop water requirements were satisfied by rain.

Approximately 500 plants were randomly uprooted using an excavator carefully avoiding damage to both canopy and root systems. The whole plants were put in sealed bags and carried to the laboratory of The Research Centre for Engineering and Agro-Food Processing of the Council for agricultural research and economics (CREA) in Monterotondo, Central Italy (42 10019″ N latitude 12 62066″ E longitude) for sampling. Firstly, the soil was removed from roots using a cold-water pressure washer. Afterwards, the plants were left to dry naturally for a few minutes. Then the roots were mechanically cut off from the plants.

The bulk density of roots was determined using a box with an internal volume of 0.0064 m^3, the value was reported as kg m^{-3}. The box was filled with roots and weighed with a KERN GmbH dynamometer (CH 50K50 model-range of measurements 50 kg and sensitivity 50 g). Three samples were taken for mean value. In 30 randomly chosen plants the fresh weight of canopy and roots were measured using a precision scale (Kern PCB 6000-0). The length of leaves and roots of this plants sample was measured with a ruler. Roots were further investigated by determining the moisture content according to [37].

2.2. Chipping, Drying and Storage

After cleaning, roots were divided into two groups (treatments) to monitor the inulin content in dried chipped roots (CRt) and dried whole roots (WRt) of cardoon in 6-months storage. CRt was obtained by selecting 15 kg of randomly chosen roots that were fresh chipped using an electric 2.0 kW bio-shredder (Zanon, mod. BIO 3). The particle size distribution (PSD) of the chipped material produced was analyzed according to [38].

Chips were collected and put into the oven for drying at 60 °C until they reached constant weight. Simultaneously, a further 15 kg of randomly chosen whole roots were collected and put in the oven for drying. At constant weight, the whole roots were removed to obtain WRt. The apparent bulk density was measured three times in CRt and WRt, respectively, according to [39] for mean value estimation.

Storage of CRt and WRt was performed outside the building, under a farm shed. Specifically, chips from CRt were collected in jute bags while the whole roots from WRt treatment were piled up as shown in Figure 1 after being labeled and weighed individually.

Figure 1. Storage under a farm shed of dried chipped roots (CRt) on the left side and dried whole roots (WRt) on the right side.

Monthly, approximately 200 g of chips from CRt and five roots from WRt were sampled and sent to ENEA laboratory for inulin determination. WRt roots were bioshredded before the shipment. A representative subsample from each treatment was kept for dry matter assessment.

2.3. Weather Data Monitoring

During the entire storage period of 6 months, the main weather-climatic parameters such as temperature, precipitation and air humidity were recorded with a weather station "DAVIS VANTAGE PRO 2" (Davis Instruments, 3465 Diablo Avenue, Hayward, CA 94545-2778, USA) located in the proximity of the storage site and connected to wireless net. Data are shown in Figure 2.

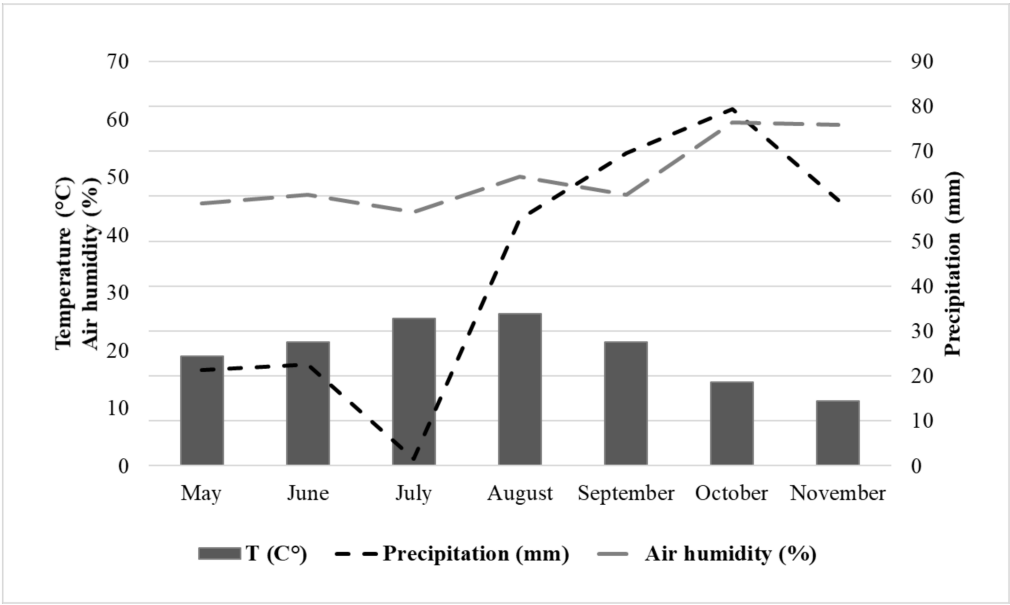

Figure 2. Trend of main climatic parameters: temperature (°C), precipitation (mm) and air humidity (%) recorded during the storage of cardoon roots from May to November 2020.

2.4. Inulin Content Determination

The collected samples from CRt and WRt were grinded to 0.5 mm in a ZM200 Retsch® ultracentrifugal mill (Retsch GmbH, Haan, Germany).

After elimination of residual humidity by drying at 50 °C for 4 h in a ventilated oven, the inulin content was determined by using a modified Raccuia method [19]. In particular, the quantitative extraction of the inulin from the roots was performed by suspending 1 g of powdered dry root in 20 mL of deionized water at 100 °C for 1 h in a Benchmark Scientific (South Plainfield, NJ, USA) Multi-Therm Heat-Shake kept under constant stirring at 500 rpm. Subsequently, the sample was centrifuged at 3500 rpm for 5 min and 4 mL of 0.75 M HCl were added to 2 mL of supernatant. The acid solution containing the inulin was then hydrolyzed in the heat-shake system for 15 min under the same conditions (100 °C, 500 rpm).

After centrifugation at 3500 rpm for 5 min, the supernatant was filtered through 0.45 µm PTFE filter (Whatman, USA) and carbohydrates were analyzed by using an HPIC DX 300 cromatographic system (Dionex, Sunnyvale, CA, USA) equipped with a Nucleogel® Ion 300 OA column (Macherey–Nagel, Düren, Germany) and sulphuric acid 10 mN as eluent. The detector was a Shodex RI101 refractive index (Showa Denko, Japan). All reagents and standards were purchased from Sigma-Aldrich (St. Louis, MO, USA). The extraction and hydrolysis processes were conducted in triplicate for each sample.

The inulin content was determined by the following Equation (1):

$$\text{Inulin } \% = \frac{\left(C_f + C_g\right) \times 0.9 \times 3}{C_R} \times 100 \quad (1)$$

where C_f and C_g are the concentration in gL^{-1} of fructose and glucose, respectively; 0.9 is the correction factor applied for the oligomer-to-monomer hydration; 3 is the dilution factor for the HCl hydrolysis; C_R is the concentration in gL^{-1} of the initial suspended roots.

2.5. Statistical Analysis

Statistical analysis was performed to assess significant differences among the mean values of dry matter and inulin content. Normality and homoscedasticity of the data were tested with Shapiro test and F test, respectively. *T*-test was performed to investigate significantly different means ($p \leq 0.05$) among treatments. Statistical analysis was performed by R 3.6.1 software to separate statistically different means [40].

3. Results and Discussion

3.1. Characterization of Cardoon Roots and Evaluation of Drying Times

The growth analysis of sample plants was performed in order to estimate the available aboveground and belowground biomass. The average fresh weight of canopy and roots was, respectively, 0.9 and 0.45 kg per plant (71 and 35 t f.w. ha^{-1}). The moisture content of roots was assessed as 70% *w/w* of fresh weight. Hence, the expected quantity of dry roots per hectare can be estimated in 10.6 t, similarly to 9.8 t DM ha^{-1} reported by [13]. Results of roots' characterization are given in Table 1.

Table 1. Characteristics of roots and canopy of three year old cardoon plants (mean ± sd) sampled in May 2020.

Taproot Length	Root Fresh Weight	Canopy Height	Canopy Fresh Weight
cm	kg	cm	kg
35.2 ± 14.9	0.45 ± 0.23	69.4 ± 6.6	0.9 ± 0.6

The bulk density of either fresh chips and dried chips was about 2.5 times higher than the bulk density of fresh whole roots and dried whole roots, respectively (Table 2).

Table 2. Bulk density of the whole roots and the chipped material, before and after drying

	Bulk Density
	(kg m^{-3})
Fresh whole roots	164.2 ± 15.9
Dried whole roots	61.79 ± 1.11
Fresh chips	418.8 ± 50.8
Dried chips	157.2 ± 31.6

Therefore, chipping represents an advisable option to reduce the volume needed for both transportation and storage. Consequently, the cost of the operations can be reduced as well. Although it was not investigated in the present study, sieving can follow the chipping phase to help removing unwanted debris from chips, which could be detrimental for further industrial processes. With this aim the chipping should be carried out with a forestry chipper able to produce a more homogeneous product.

On the contrary, in our case, as shown by PSD analysis (Figure 3), 90% of the chipped material was less than 8 mm length, making it not possible to separate by sieving unwanted debris from chips.

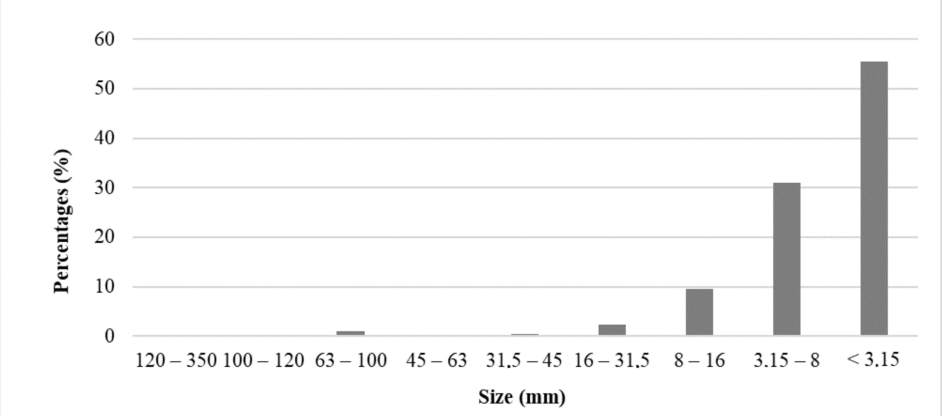

Figure 3. Particle size distribution of the chipped roots.

Moreover, the drying process can also benefit from chipping by reducing time and energy required [41]. According to our results, indeed, chipped roots could reach constant weight after 48 h, whilst whole roots needed 24 h more to dry completely (Figure 4).

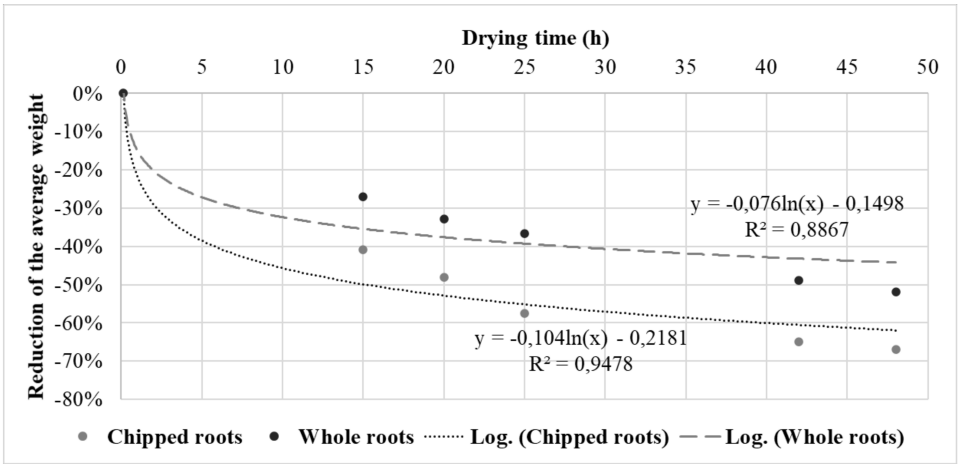

Figure 4. Reduction of the average weight of whole roots and chipped roots during drying in a thermo-ventilated oven at 60 °C.

3.2. Inulin Content

Inulin content at T0 (beginning of storage, immediately after drying) was 43.5 ± 0.65% and 47.1 ± 1.30% *w/w* in WRt and CRt, respectively (Figure 5). Drying time negatively affected the inulin content in WRt which resulted in 3.54% *w/w* lower than CRt. Conceivably, in WRt treatment, the inner tissues of the roots took longer to dry out than the outmost tissues of the same root. Hence, the metabolic activities naturally occurring in living cells stopped later and this partially explains the loss of inulin in WRt.

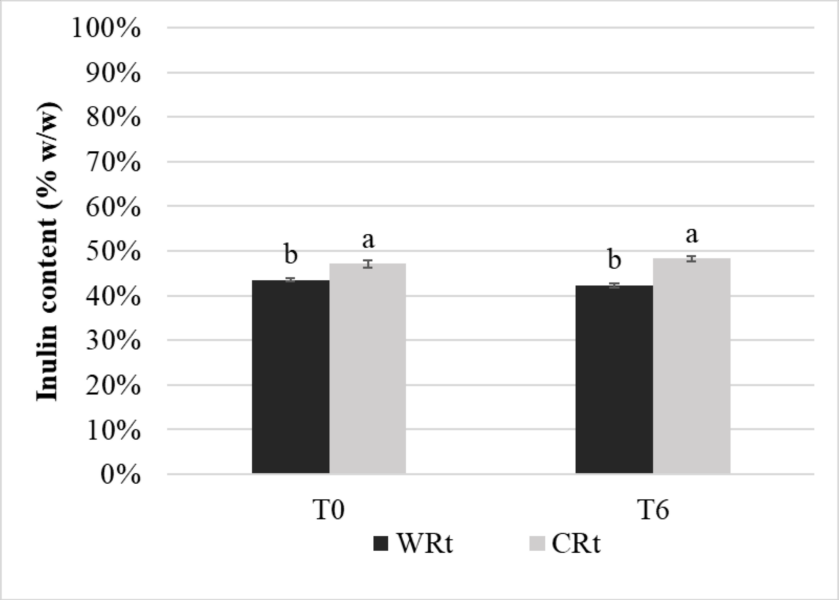

Figure 5. Inulin content in dry whole and chipped roots of cardoon at T0 and after 6 months of storage (T6). Common letters denote the absence of significant difference ($p < 0.05$).

Regardless of the difference in inulin content found among treatments, the values herein reported are consistent with the literature although some authors also highlight that possible changes in inulin content may be experienced according to the harvest season [14]. Higher values are usually found in spring, particularly between full blossom and fruit ripening. For this reason, root sampling was performed in May when the concentration is supposed to peak.

After 6 months storage (T6), inulin content did not change significantly in comparison with the initial content measured in the respective treatment, namely: 42.3 ± 0.82% *w/w* in WRt and 48.3 ± 1.12% *w/w* in CRt. Therefore, the drying process performed before the storage prevented the degradation of inulin, at least over the following 6 months of storage. This finding highlights the possibility for industries to exploit the drying process at the industrial scale to help storing the cardoon roots for longer time since inulin loss is prevented effectively. Artificial drying is certainly costly in terms of money and energy, but it is surely less costly than freezing. Additionally, the machinery required for drying is easier to run and cheaper to buy (e.g., a ventilated oven) with enormous advantage also for the transportation which does not longer require the ice-chain. Chipping also contributes to enhance the inulin supply chain as the higher bulk density of chipped material would require less space for drying, storage and transportation.

3.3. Moisture Content and Dry Matter Content

The moisture content at the first month of storage (T1) in WRt and CRt increased significantly up to 12.1% and 10.8%, respectively. This was probably due to the reabsorption of humidity from the external environment. In fact, as shown in Figure 6, the monthly moisture content measured in WRt and CRt traces the air moisture pattern recorded by weather station. The moisture increase in roots was more evident in WRt where 16.3% *w/w*

of moisture was recorded at T6 (i.e., 3.4% higher than CRt). This was probably due to a greater exposure of the whole roots to air humidity with respect to the chipped material stored inside the jute bag.

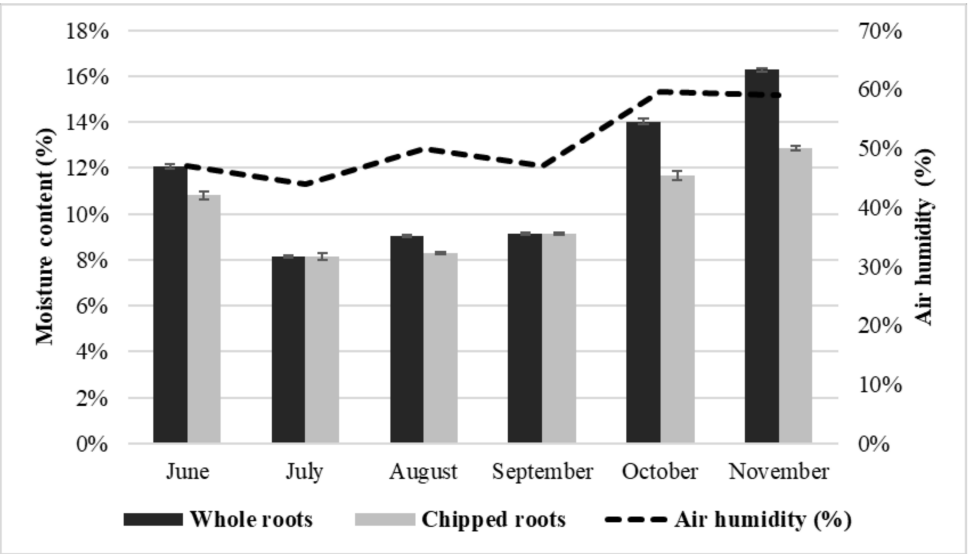

Figure 6. Moisture content (mean ± SD) in WRt and CRt during the 6 months storage in comparison with air moisture recorded by a weather station over the same period.

During the first month of storage, a significant reduction in weight in both treatments was recorded: 10.9% and 3.5% in WRt and CRt, respectively (Figure 7). Despite this, during the following months the roots' dry weight remained constant in both treatments. However, a significant difference of approximately 6% was recorded between the treatments throughout the trial. Both results were probably due to the higher water content recorded in the whole roots—not completely removed after drying (less drying efficiency) or greatly reabsorbed from the external environment (higher exposure to air humidity)—which promoted microbial activity.

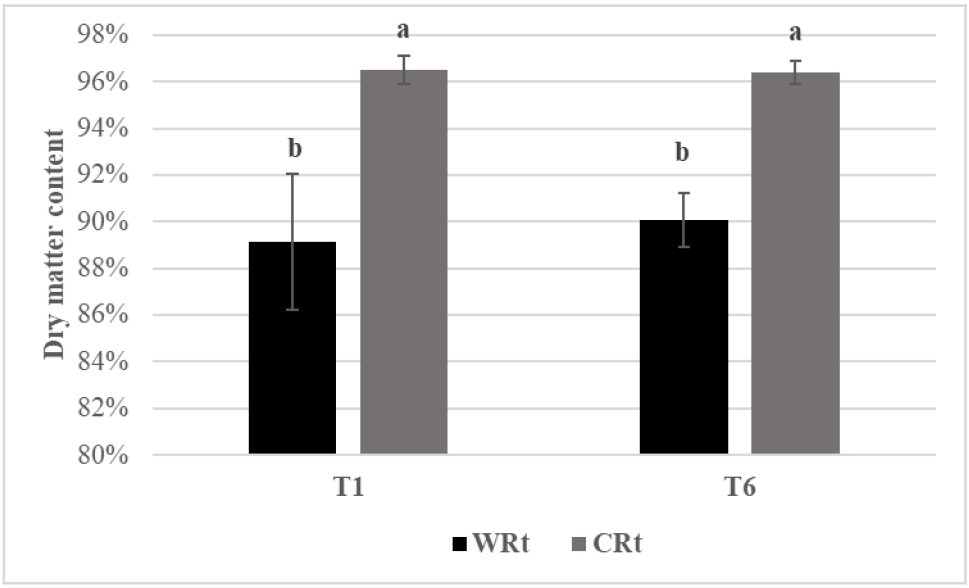

Figure 7. Dry matter content in dried whole roots (WRt) and dried chipped (CRt) roots at the first month of storage (June, T1) and after 6 months of storage (T6). Common letters denote the absence of significant difference ($p < 0.05$).

4. Conclusions

In the perspective of new generation biorefineries and the circular bioeconomy framework, the exploitation of cardoon also for inulin production is rather appealing, particularly if plants have been previously exploited for the production of further high added-value raw materials like seeds and stalks. Due the limited favorable period for harvesting the roots when inulin content is maximum, industries need to store enormous quantities of roots and process them gradually. Hence, storage plays a fundamental role in supply chain. Our findings suggest that during a 6-month storage inulin loss is negligible if roots are previously dried. Furthermore, chipping could also be a good practice since it is possible to reduce the volume required for storage (and also transportation) while it promotes a quicker drying; thus, less energy is required to dry out the roots.

In conclusion, our results highlight the possibility to chip cardoon roots meant for inulin extraction to ameliorate the supply chain of such a material. Although drying remains a costly strategy, chipping would help to reduce such cost by reducing the time required. However, further studies should provide clues to improve also the harvesting and cleaning process.

Author Contributions: Conceptualization, L.P. and V.A.; methodology, V.A., W.S. and F.L. (Francesco Latterini); validation, A.C.; formal analysis, V.A., W.S., F.L. (Francesco Latterini), F.L. (Federico Liuzzi) and V.V.; investigation, V.A., W.S., F.L. (Francesco Latterini), F.L. (Federico Liuzzi) and V.V.; resources, A.C.; writing—original draft preparation, V.A., F.L. (Federico Liuzzi), V.V.; writing—review and editing, V.A., W.S. and F.L. (Francesco Latterini).; supervision, L.P. and I.D.B.; funding acquisition, L.P. and I.D.B. All authors have read and agreed to the published version of the manuscript.

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Data Availability Statement: Data is not publicly available, though the data may be made available on request from the corresponding author.

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Conflicts of Interest: The authors declare no conflict of interest.

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