Data Article

$^1$H NMR metabolic profiling dataset of spiny chicory ($Cichorium$ $spinosum$ $L.$) exposed to abiotic stresses

Georgia Ntatsi$^{a,*}$, Konstantinos A. Aliferis$^{b,c,*}$, Angeliki Panagiotopoulou$^d$, Youssef Rouphael$^e$, Dimitrios Savvas$^a$

$^a$ Laboratory of Vegetable Production, Department of Crop Science, Agricultural University of Athens, Iera Odos 75, 11855 Athens, Greece
$^b$ Laboratory of Pesticide Science, Department of Crop Science, Agricultural University of Athens, Iera Odos 75, 118 55, Athens, Greece
$^c$ Department of Plant Science, McGill University, Macdonald Campus, Ste-Anne-de-Bellevue, QC H9X 3V9, Canada
$^d$ Institute of Biosciences & Applications, NCSR “Demokritos”, Aghia Paraskevi, 15310 Attiki, Greece
$^e$ Department of Agricultural Sciences, University of Naples Federico II, Portici, Italy

Article history:
Received 12 February 2020
Accepted 20 April 2020
Available online 28 April 2020

Keywords:
Isosmotic solution
Functional foods
Hydroponics
Plant metabolomics
Salinity stress
Stamnagathi

Abstract

The data presented here were derived by $^1$H NMR metabolic profiling of stamnagathi ($Cichorium$ $spinosum$ $L.$) plants following treatments with different isosmotic salt solutions; eight saline nutrient solutions with two different levels of total molar concentrations, which were obtained by adding different amounts of NaCl, KCl, Na$_2$SO$_4$ or CaCl$_2$ to the replenishment nutrient solution, were applied. The $^1$H NMR metabolite profiles of stamnagathi plants, which are included in this article, were recorded 56 days after transplanting. Since stamnagathi is a niche product combining unique taste and superior phytonutrient content (e.g. vitamins C and K1, lutein, $\beta$-carotene, tocopherols, phenolic acids, fatty acids, minerals, and glutathione), the dataset could serve as a reference for future metabolomics studies related to the investigation of the effects of the four salinity sources on the plant’s metabolism. Also, the dataset could be a valuable resource for the discovery of validated biomarkers of the plant’s tolerance to salinity stress and responses to new plant protection products (e.g. bioelicitors). The

* Corresponding authors.
E-mail addresses: ntatsi@aua.gr (G. Ntatsi), konstantinos.aliferis@aua.gr (K.A. Aliferis).

https://doi.org/10.1016/j.dib.2020.105622
2352-3409/© 2020 Published by Elsevier Inc. This is an open access article under the CC BY-NC-ND license.
(http://creativecommons.org/licenses/by-nc-nd/4.0/)
Specifications Table

| Subject | Agricultural and Biological Sciences |
| Specific subject area | Plant metabolomics |
| Type of data | $^1$H NMR spectra (*.jdx format) |
| How data were acquired | $^1$H NMR spectra metabolomics analysis |

Instrument: 11.7 T Bruker Avance DRX spectrometer at 500 MHz equipped with a 5 mm inverse detection probe

Acquisition of data using the software TopSpin 1.3 (Bruker, Germany)

Raw JCAMP (*.jdx)

NMR Analysis

Locked on the deuterium signal of the D2O
Temperature, T=298 K
Number of scans, ns=128
Acquisition time: 2.6 s
Relaxation delay: 2 s
90° pulse length of 8 μs
Spectral width, sw=12 ppm, SWH= 6000.615 Hz
Presaturation of H$_2$O during the recycle delay

NMR Data Processing

Fourier transformation
Interactive phase correction
Baseline correction
Exponential line-broadening window function, lb=0.3 Hz
Trimethylsilyl-2,2,3,3-d$_4$-propionic acid sodium salt (TSP) reference signal set at 0.0 ppm

Normalization of NMR Bin Data

0.04 ppm bin size

Description of data collection

$^1$H NMR metabolite profiling (*.jdx format)

Data source location

Agricultural University of Athens

Athens

Greece

Data accessibility

Repository name: Pesticide Metabolomics Group data repository

Data identification number: Cichorium spinosum (PMG-01-20)

Direct URL to data:

https://www.aua.gr/pesticide-metabolomicsgroup/Resources/libraries/Cichorium_spinosum_(PMG-01-20)_data_set/Cichorium_spinosum_(PMG-01-20).html

Related research article

Salinity source alters mineral composition and metabolism of Cichorium spinosum. Environmental and Experimental Botany (141, 113-123). DOI; http://dx.doi.org/10.1016/j.envexpbot.2017.07.002 [1]

Value of the data

- The data provide an overview of the effects of four sodium and chloride salts (Na$_2$SO$_4$, NaCl, KCl or CaCl$_2$) on the metabolism of C. spinosum grown in a closed soilless cultivation system
- The dataset could be used by researchers working on the study of the effects of different iso-osmotic salinity levels on the metabolism of model biological systems
- The $^1$H NMR metabolite profiles could serve to further support the cultivation of C. spinosum as a functional food
- To the best of our knowledge, no similar data exist on the effects of the four salinity sources on the metabolism of stamnagathi

dataset support the research article “Salinity source alters mineral composition and metabolism of Cichorium spinosum” authored by Ntatsi et al., (2017) [1].

© 2020 Published by Elsevier Inc.

This is an open access article under the CC BY-NC-ND license. (http://creativecommons.org/licenses/by-nc-nd/4.0/)
1. **Data Description**

Processed $^1$H NMR metabolite profiles (*.jdx format) of *Cichorium spinosum* L. (stamnagathi, Asteraceae) corresponding to profiles of untreated (control) plants and plants treated with Na$_2$SO$_4$, NaCl, KCl or CaCl$_2$ at two different iso-osmotic levels. Analyses were performed in leaf samples collected 56 days after transplanting.

2. **Experimental Design, Materials and Methods**

2.1. **Plant cultivation and experimental treatments**

Spiny chicory (*C. spinosum* L.), also known in Greek language as stamnagathi, was used to monitor the effects of Na$_2$SO$_4$, NaCl, KCl or CaCl$_2$ at two different iso-osmotic levels on its metabolism and the discovery of the corresponding biomarkers of stress [1]. The experiment was conducted in a glasshouse at the Agricultural University of Athens. Seeds of stamnagathi originating from Crete, were sown in seed trays, and at the stage of three true leaves were transferred into 36 closed-loop hydroponic circuits (experimental plots). The treatments were consisted of nine nutrient solutions (NSs); a basic NS served as control, and eight saline NSs, with two different levels of total molar concentrations, which were obtained by adding different amounts of NaCl, KCl, Na$_2$SO$_4$ or CaCl$_2$ to the replenishment NS. [1]. The experimental design and sample preparation was based on previously described protocols following optimization [2–3].

2.2. **Sampling and metabolite extraction**

Leaf tissues of *C. spinosum* L. of the same physiological age were pulverized to a fine powder under liquid N$_2$, and a portion (100 mg of fresh weight, FW) was lyophilized for 24 h. For the extraction of the polar metabolite fraction, 1 mL of deuterium oxide (D$_2$O) containing 0.05% trimethylsilyl- 2,2,3,3-d$_4$-propionic acid sodium salt (TSP) (Sigma-Aldrich Chemie GmbH, Munich, Germany) was added in the lyophilized leaf tissues. The resulting suspensions were sonicated for 25 min and then, they were kept under continuous agitation (150 rpm) (1 h at 24°C). For the removal of debris, the suspensions were centrifuged (12,000g) for 1 h at 4°C and the supernatants were subjected to a second centrifugation (12,000g) for 30 min at 4°C. The supernatants were then collected and kept in Eppendorf tubes at −80°C until the acquisition of $^1$H NMR spectra.

2.3. **$^1$H NMR analysis**

Extracts were placed in NMR tubes (5 mm Thin Wall Precision NMR Sample Tubes 8” L, Wilmad, Vineland, NJ, USA) for the recording of $^1$H NMR spectra. A Bruker Avance DRX spectrometer (500 MHz) equipped with a 5 mm inverse detection probe at 298 K, was employed in analyses. NMR parameters and the magnetic field homogeneity were optimized using a control stamnagathi plant extract. A total of 128 transients of 64 K data points were acquired per sample. The acquisition time was set at 2.6 s and a relaxation delay of 2 sec was inserted into the pulse sequence. A 90° pulse with water pre-saturation sequence was applied.

2.4. **Data pre-processing and biomarker discovery**

The pre-processing and deconvolution of the obtained spectra, multivariate analyses, and biomarker discovery were performed as previously described [2–3], with minor modifications.
Initially, spectra were Fourier transformed and their phase and baseline were automatically corrected. The offsets of chemical shifts were corrected based on the signal of TSP at 0.00 ppm using the software Spectrus Processor (ACD Labs, Toronto, Canada). The metabolite identification was based on chemical shifts, coupling constants ($J$), and comparisons to $^1$H NMR spectra of analytical standards of selected plant metabolites in D$_2$O that had been acquired using the same analyser operating under identical analytical conditions. Additionally, the ACD/C+H NMR Predictor and DB function of the Spectrus Processor and information from the literature were used for the annotation of unknown shifts. The spectral region between 0.70 and 8.80 ppm was integrated after the removal of regions such as, the one that corresponds to the water signal (4.70–4.90 ppm), using the “intelligent bucketing” option of the software and bin size equal to 0.04 ppm.

2.5. Experimental data analysis

The discovery of trends within the obtained dataset and the discovery of the biomarkers of stamnagathi’s response to the different salinity levels and sources, was based on multivariate analyses [2–3].

Conflict of Interest

The authors declare no conflict of interest.

Supplementary material

Supplementary material associated with this article can be found, in the online version, at doi:10.1016/j.dib.2020.105622.

References

[1] G. Ntatsi, K.A. Aliferis, Y. Rouphael, F. Napolitano, K. Makris, G. Kalala, G. Katopodis, D. Savvas, Salinity source alters mineral composition and metabolism of Cichorium spinosum, Environ Exp Bot 141 (2017) 113–123 https://doi.org/10.1016/j.envexpbot.2017.07.002.

[2] K.A. Aliferis, R. Chamoun, S. Jabaji, Metabolic responses of willow (Salix purpurea L.) leaves to mycorrhization as revealed by mass spectrometry and $^1$H NMR spectroscopy metabolite profiling, Front Plant Sci 6 (2015) 344 https://doi.org/10.3389/fpls.2015.00344.

[3] K.A. Aliferis, S. Materzok, G.N. Paziotou, M. Chrysayi-Tokousbalides, Lemma minor L. as a model organism for ecotoxicological studies performing $^1$H NMR fingerprinting, Chemosphere 76 (2009) 967–973, doi:10.1016/j.chemosphere.2009.04.025.