Data Article

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

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A R T I C L E   I N F O

A B S T R A C T

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, β-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

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

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)
Data format	Raw JCAMP (*.jdx)
Parameters for data collection	**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_2O 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-d4-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-metabolicsgroup/Resources/libraries/ Chichorium_spinosum_(PMG-01-20)_data_set/Chichorium_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_2SO_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

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_2SO_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_2SO_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_2SO_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 lyophylized for 24 h. For the extraction of the polar metabolite fraction, 1 mL of deuterium oxide (D_2O) containing 0.05% trimethylsilyl- 2,2,3,3-d4-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 90º 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_2O 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

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