Data Article

Dataset on functional and chemical properties of the medicinal halophyte *Polygonum maritimum* L. under greenhouse cultivation

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**ABSTRACT**

This data article includes data and analyses on the effect of different agronomic techniques on the production of *Polygonum maritimum* L. (sea knotgrass), namely different salinity irrigation treatments (0, 100, 200, 300 and 600 mM of NaCl) and a multi-harvest regime, and their relation with the chemical profile (ultra-high-resolution mass spectrometry - UHRMS), *in vitro* antioxidant [radical-scavenging activity (RSA) of DPPH and ABTS, copper chelating activity and ferric reducing antioxidant power] and anti-inflammatory (nitric oxide reduction on lipopolysaccharide-stimulated macrophages) activities. For further interpretation of the data presented in this work, please see the related research article “The irrigation salinity and harvesting affect the growth, chemical profile and biological activities of *Polygonum maritimum* L.” (Rodrigues et al., 2019).

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1. Data

The sea knotgrass plants were produced in a greenhouse under different irrigation conditions (freshwater, 100, 200, and 300 mM of NaCl), and submitted to three consecutive harvests. Obtained biomass (above ground organs) were extracted with acetone, and the extracts were tested for in vitro antioxidant (radical-scavenging activity (RSA) of DPPH and ABTS, copper chelating activity (CCA) and ferric reducing antioxidant power (FRAP)) and anti-inflammatory (nitric oxide reduction on lipopolysaccharide-stimulated macrophages) properties. The results of half maximal inhibitory concentration (IC50) are reported in Tables 1 and 2, for antioxidant and anti-inflammatory, respectively. For the same treatment, the RSA towards DPPH and ABTS, and CCA increased with the harvest. The same tendency was observed in FRAP, except on freshwater-irrigated plants that showed decreased activity from 1st to 2nd harvest, however decreasing in the 3rd harvest. The anti-inflammatory activity decreased with the harvest, and the lowest IC50 values were obtained on biomass from the 1st harvest, for all treatments. A detailed chemical profiling was performed by LC-UHRMS [1] and differences between treatments and harvests were analysed by PCA and PLC-DA statistics (Figs. 1–3). Striking differences on the chemical composition of statistically significant peaks tends to differ along with consecutive harvests and showed clear separation of salt concentration treatments disregarding the harvest sequence.

2. Experimental design, materials, and methods

2.1. Extraction

The dried biomass of aerial parts (leaves, stems and shoots) was extracted with pure acetone (1:40, w/v) in an ultrasonic bath [1]. The extracts were filtered (Whatman no. 4) and acetone was removed by
rotary evaporation. The dried extracts were weighed, resuspended in methanol at 10 mg/mL, and stored at \(-20^\circ\text{C}\).

2.2. Radical-scavenging activity (RSA) on DPPH and ABTS

The DPPH and ABTS RSA of the extracts at different concentrations (10–1000 \(\mu\text{g/mL}\)) was performed as reported earlier [2]. Differences in absorbance were measured in a microplate reader (Biotek Synergy 4). Butylated hydroxytoluene (BHT) was used as standard at concentrations equal to those of the samples. Results were expressed as an inhibition percentage, comparative to a control containing methanol instead of the sample, and as half maximal inhibitory concentration (IC\(_{50}\) values, \(\mu\text{g/mL}\)).

2.3. Ferric reducing antioxidant power (FRAP)

The extracts’ capacity to reduce Fe\(^{3+}\) (at concentrations amongst 10–1000 \(\mu\text{g/mL}\)) was evaluated as described by Rodrigues et al. [2]. An increase in the absorbance at 700 nm in the reaction mixture indicates an increased reducing power of the samples (Biotek Synergy 4). Results were calculated as a percentage in relation to the standard (BHT, 1000 \(\mu\text{g/mL}\)), and as IC\(_{50}\) values (\(\mu\text{g/mL}\)).
2.4. Metal chelating activity on copper (CCA)

The CCA of the extracts (at concentrations varying between 10 and 1000 μg/mL) was assayed as depicted before [2]. The color switch was measured on a microplate reader (Biotek Synergy 4), and ethylenediaminetetraacetic acid (EDTA) was applied as the positive control at the identical concentrations of the extracts. Results were presented as an inhibition percentage comparatively to a control using methanol as substitute of the sample, and as IC50 values (μg/mL).

2.5. Cell culture and cell viability

RAW 264.7 cells were grown in RPMI 1640 culture medium complemented with 10% heat-inactivated fetal bovine serum (FBS), 1% L-glutamine (2 mM), and 1% penicillin (50 U/mL)/streptomycin (50 μg/mL) and were kept at 37 °C in moistened environment with 5% CO2. Cells were seeded at a

![Fig. 1](image-url)
concentration of 1 \times 10^4 \text{ cells/well}, in 96-well microplates. After 24h of incubation, the extracts were added at concentrations from 3 to 100 \text{ mg/mL}, and incubated for 24h. Cells treated with the vehicle (0.5% DMSO, v/v) were used as negative control, and cell viability was assessed through the 3-(4,5-dimethylthiazol-2-yl)-2,5-diphenyltetrazolium bromide (MTT) colorimetric test \cite{3}. Results were calculated as a percentage of cell viability, in comparison with the control cells.

2.6. In vitro anti-inflammatory assay

The samples were tested for their capacity to decrease nitric oxide (NO) production in RAW 264.7 macrophages \cite{3}. Cells were plated at 2.5 \times 10^5 \text{ cells/well} in 96-well plates and left to adhere overnight. Then, non-toxic concentrations of the extracts (>80% of cell viability) were incubated in serum- and phenol-free culture medium, with 100 ng/mL of LPS, for 24h. The cellular NO production was evaluated.
by the Griess method [3]. Results were expressed as a percentage (%) of NO production comparing to a control cells containing DMSO (0.5%, v/v), and as IC₅₀ values (µg/mL).

2.7. Liquid chromatography/ultra-high-resolution mass spectrometry (LC-UHRMS)

Samples were pre-treated using solid phase extraction as follows: 100 mg of the extracts were suspended in 1 mL of 0.2% formic acid in purified water (HLP10UV, Hydrolab, Gdańsk). Next, the suspension was loaded to the C18 Sep-Pak cartridges (1 cm³, 360 mg, Waters Corp., Milford, MA) and washed with 0.5% methanol to remove carbohydrates and then with 80% methanol to elute phenolics. The phenolic fraction was re-evaporated, dissolved in 1 mL of 0.2% formic acid in 80% aqueous methanol, centrifuged for 5 min at 23 000 × g, and filtered through 0.22 µm syringe filters (mix cellulose
esters, Carl Roth, Karlsruhe, Germany) before LC-MS analysis (stored at −20 °C before analysis for no longer than 3 days). All analyses were performed in triplicate for three independent samples.

Liquid chromatography (LC) – electrospray ionization (ESI)-QTOF-MS was carried out using Thermo Dionex Ultimate 3000 RS system consisting of a binary pump system, sample manager, column manager and a DAD detector (Thermo Fischer Scientific, Waltham, MA), coupled to a Bruker Compact quadrupole time-of-flight (QTOF) mass spectrometer (Bruker Daltonics, Billerica, MA). Separations were performed on a Kinetex C18 column (2.1 × 100 mm, 2.6 μm, Phenomenex, USA), with mobile phase A consisting of 0.1% (v/v) formic acid in water and mobile phase B containing 0.1% (v/v) formic acid in acetonitrile. A linear gradient from 1% to 60% phase B in phase A over 20 minutes was used to separate phenolic compounds. The flow rate was 0.4 mL/min, and the column was held at 30 °C. Mass spectra were acquired in negative-ion mode with 5 Hz frequency over a mass range from m/z 100 to 1500. Operating settings of the ESI ion source were as follows: capillary voltage 3 kV, dry gas flow 6 L/min, dry gas temperature 200 °C, nebulizer pressure 0.7 bar, collision radio frequency 700.0 V, transfer time 100.0 μs, and pre-pulse storage 7.0 μs. Ultrapure nitrogen was used as drying and nebulizer gas, and argon was used as the collision gas. The collision energy was set automatically from 15 to 75 eV depending on the m/z of the fragmented ion. For calibration of the accurate mass measurements, we used sodium formate introduced to the ion source at the beginning and end of each separation via a 20 μL loop. After data acquisition, raw UPLC–QTOF–MS spectra (negative mode) were pre-processed using a ProfileAnalysis software (version 2.1, Bruker Daltonik GmbH, Germany). Parameters of ProfileAnalysis were used as follows: advanced bucket generation with retention time range of 0–20 min, mass range of 100–800 m/z, each bucket (spectral bins) was formed with 1 min and 1 m/z delta, 0.2 kernelizing value, without normalization, background subtraction, and time alignment. LC-MS analyses were processed with the Find Molecular Futures (FMF) function to create compounds (molecular features) with S/N- 3 for peak detection. Generated bucket table consisting of tR:m/z pairs and respective compound intensity was exported and uploaded to MetaboAnalyst program. Each obtained dataset was filtered and normalized to the sum of peak areas and mean-centered scaling.

Acquired spectra were processed with Bruker DataAnalysis 4.3 software. The quality of the isotopic fit was expressed by the mSigma-value. The matched peaks from SmartFormula3D were sent to MetFrag website for computer-assisted in silico fragmentation and identification of metabolite mass spectra. Additionally, we searched the web-based databases for potential matches to the detected compounds: the human metabolome database (http://www.hmdb.ca/), the BiGG database (http://bigg.ucsd.edu/), the PubChem database (http://pubchem.ncbi.nlm.nih.gov/), the MassBank database (http://www.massbank.jp), KEGG (www.genome.jp) and the Metlin database (http://metlin.scripps.edu).

2.8. Statistical analysis

Results were expressed as the mean ± standard error of the mean (SEM) of at least three repetitions. Significant differences were evaluated by analysis of variance (ANOVA) and by the Tukey HSD test (P < 0.05). Statistical analyses were made using the XLSTAT statistical package for Microsoft Excel (version 2013, Microsoft Corporation). The IC50 values were calculated by a sigmoidal fitting of the data (GraphPad Prism v. 5.0 program).

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Conflict of interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.dib.2019.104357.

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