Metabolite quantification data based on 1H-NMR profiling of eggplant or pepper fruit during its development

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DATA NOTE

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Metabolite quantification data based on ^{1}H-NMR profiling of eggplant or pepper fruit during its development

Léa Roch[1], Catherine Deborde[1,2,3,4], Daniel Jacob[1,2,3,4], Anaïs Clavé[1], Marguerite Batsale[1], Yves Gibon[1,2] and Annick Moing[1,2*]

Abstract

Objectives The primary metabolite contents of ripe fruits result from complex regulations during their development. For Solanaceae, these regulations have been widely studied in tomato. The fruit metabolite contents of other fruit species, such as pepper (*Capsicum annuum* L.) and eggplant (*Solanum melongena* L.), constitute a valuable resource for the community to study the regulation of fruit metabolism and identify common or species-dependent regulations. This dataset about major polar metabolites is part of a larger project that integrates other omics data for pepper and eggplant, and other fruit species for metabolomics and other omics.

Data description We provide quantitative metabolite data of pepper and eggplant fruit along development. We sampled pepper and eggplant fruit cultivated in a tunnel or a greenhouse at 10 or 11 stages from anthesis to ripe fruit. We used proton nuclear magnetic resonance (^{1}H-NMR) metabolomic profiling of polar extracts to quantify the major metabolites and expressed the data in µmol per g fresh weight. Twenty-four metabolites were determined in pepper and 27 in eggplant. Nineteen common metabolites were quantified in both fruit species including three soluble sugars and one sugar-alcohol, five organic acids and nine free amino acids. These data can be combined with similar quantitative data on other species or complemented with other omics data to perform cross-species or cross-omics comparisons.

Keywords Fruit, Metabolomics, Quantification, Proton NMR, *Capsicum annuum* L., *Solanum melongena* L.

Objective

To obtain an integrated vision of fruit development and related metabolism, we studied several species with the same approach. This dataset is part of a larger study about eight fleshy fruit species aiming at studying the regulation of fruit metabolism during fruit development by combining several omics. During this project, special care has been taken to produce quantitative data whenever possible, on fruits sampled at a range of developmental stages before and during ripening, as already done for tomato [1, 2]. Such quantitative data are of special interest for analyses and meta-analyses aiming at showing common or species-dependent regulations. For metabolomics, when certain precautions are taken during extraction,

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spectra acquisition and processing, and using calibration, proton nuclear magnetic resonance (^{1}H-NMR) profiling provides absolute quantification data of major metabolites [3] expressed for instance as mg or mmol on a fresh weight or dry weight basis. The present quantitative data of the major metabolites of both eggplant (*Solanum melongena* L.) and pepper (*Capsicum annuum* L.) fruit based on monodimensional (1D) ^{1}H-NMR spectra have not been published in a research paper yet. When complemented with other metabolomics data and with other omics they can be used for a systems biology study [4] to decipher and improve fleshy fruit quality [5, 6].

Data description

Plant material

The plants were grown under conditions of commercial production at Sainte-Livrade-sur-Lot (South-West France, 44° 23′ 56″ N, 0° 35′ 25″ E, 50-m altitude). Pepper plants, *C. annuum* cv Gonto (Clause Vegetable Seeds, Portes-lès-Valence, France), were grown under a plastic tunnel at 1.8 plant/m^2 density in a sandy-loam soil with drip fertirrigation (2 to 3 irrigations per day during 15–20 min with a flow rate of 4.5 mm/h/m^2). Eggplant plants, *S. melongena* cv Monarca (Rijk Zwaan, Aramon, France), were grown in a plastic greenhouse at 1.2 plants/m^2 density in coco-fiber substrate with drip fertirrigation (irrigation every 60 to 80 min from 8:30 a.m. to 5:30 p.m. triggered by a solarimeter during 6–7 min with a dripper flow rate of 2 L/h, five drippers for three plants and an electrical conductivity sensor for fertilizer monitoring). From anthesis to ripe-fruit harvest, the mean, minimum and maximum daily-mean temperatures were 22.9, 17.0, 28.9, and 22.8, 17.3, 27.6 °C, for pepper in the tunnel and eggplant in the greenhouse, respectively. Fruits were harvested at ten or 11 stages of development (Data files 1–2, [7] and [8], respectively): anthesis, growth stages, maturation start, ripening stages, ripe). Fruit harvests started on June 20th and May 27th and ended on October 5th and August 11th 2016 for pepper and eggplant, respectively. Each stage was identified with its corresponding number of days after anthesis (DPA). Five biological replicates were collected for each stage of development, with a minimum of 12 fruits per replicate for the first two stages and four fruits for the other stages (Data files 3–4 [9, 10]). For the first two stages, the entire ovary or fruit was sampled as their rapid dissection was not feasible. For the following stages, the seeds were discarded to study the edible fleshy part of the fruit. Then, for pepper samples were dissected from about one third of the fruit around the equatorial region, and for eggplant from about one fourth of the fruit on the pedicel side. Pepper fruit pericarp or eggplant fruit mesocarp (pericarp without peel) was rapidly dissected. All samples were immediately frozen in liquid nitrogen, stored at − 80 °C before cryogrinding (Spex Genogrinder 2010, Fisher Scientific, Illkirch, France) and lyophilization (Dura Dry MP Freeze Dryer, Warminster, PA USA), and then NMR-based analysis.

Proton NMR profiling data

Polar compounds were extracted from 25 ± 1 mg lyophilized powder with an ethanol–water series [11]. NMR analyses of major polar compounds were performed on pH-adjusted lyophilized extracts as previously described [11, 12] with minor modifications (Data file 5 [9]). Briefly, absolute quantification of individual metabolites was achieved using a 500-MHz Avance-III NMR spectrometer (Bruker Biospin, Wissembourg, France) and external calibration with calibration-range solutions. The NMR spectrometer was equipped with a 5-mm inverse probe and an autosampler (Bruker Biospin, Karlsruhe, Germany). ^{1}H-NMR spectra were acquired with a single pulse (zg) sequence, 64 scans, a 2.73-s acquisition time, a 90° pulse angle, a 25-s recycle delay and a fixed receiver gain for each species. The resulting free induction decays (Data sets 1–2 [9, 10]) were processed with NMRProcFlow tool [13] using the variable-size bucketing module for peak integration.

Metabolites were assigned according to published data [14], previous work on a mixture of stages of development [12], and additional 1D and 2D NMR experiments including COrrelation SpectroscopY (COSY), Heteronuclear Multiple Bond Correlation (HMBC), Heteronuclear Single Quantum Correlation (HSQC), 1D selective gradient COSY and TOtal Correlation SpectroscopY (TOCSY) (1D ^{1}H annotation Table and 2D spectra in Data sets 3–4 [9, 10]). For pepper, two unknown compounds were partially identified: Unknown_1, a trans-4-hydroxyproline like compound, and Unknown_2, a hydroxycinnamic-acid containing compound (Data set 5 [9]). The singlet at 3.05 ppm from Unknown_1 was infirmed to be malonate or creatine in disagreement with previous studies [15, 16]. A resonance group was selected for metabolite quantification (assignment description Data files 6–7 [7, 8], localization on representative 1D spectra Data files 8–9 [9, 10]). Metabolite contents were determined using the calibration curves and the dry matter contents of the samples and expressed on a fresh weight basis. This resulted in the quantification of 24 and 27 metabolites in pepper and eggplant, respectively (Data files 10–11 [7, 8], overview with principal component analyses (PCA) Data files 12–13). Nineteen metabolites were determined in both pepper and eggplant, including three soluble sugars, five organic acids and nine free amino acids. These common metabolites allowed seeing common changes during development for the two species and their main compositional differences (PCA Data file 14 [7, 8]).

The strategy for spectra, data and metadata deposit combines a national repository (recherche.data.gouv, https://

Table 1 Overview of data files, data sets or data subsets

Label	Name of data file/data set-subset	File types (file extension)	Data repository and identifier (DOI or accession number)
Data file 1	Pepper-Sampling-Figure.pdf	Figures (pdf)	ODAM (https://identifiers.org/odam.explorer:FR17PP009) [7]
Data file 2	Eggplant-Sampling-Figure.pdf	Figures (pdf)	ODAM (https://identifiers.org/odam.explorer:FR17EP006) [8]
Data file 3	1_Pepper-Sample-Metadata.csv	Comma separated text files (.csv)	recherche.data.gouv (https://doi.org/10.57745/U9K1BB) [9]
Data file 4	1_Eggplant-Sample-Metadata.csv	Comma separated text files (.csv)	recherche.data.gouv (https://doi.org/10.57745/AXXEXR) [10]
Data file 5	0_Pepper-NMR-Methods.pdf 0_Eggplant-NMR-Methods.pdf	Text (.pdf)	recherche.data.gouv (https://doi.org/10.57745/U9K1BB) [9] recherche.data.gouv (https://doi.org/10.57745/AXXEXR) [10]
Data set 1	Raw FID and processed ^{1}H-NMR spectra of pepper extracts (MBBBI_18P08-LR_Pepper.zip) and NMRProcFlow processing scripts	zip files (.zip, TopSpin Bruker format when unzipped) Text files (.txt)	recherche.data.gouv (https://doi.org/10.57745/U9K1BB) [9]
Data set 2	Raw FID and processed ^{1}H-NMR spectra of eggplant extracts (ACBBI_17P05-LR_Eggplant.zip) and NMRProcFlow processing scripts	zip files (.zip, TopSpin Bruker format when unzipped) Text files (.txt)	recherche.data.gouv (https://doi.org/10.57745/AXXEXR) [10]
Data set 3	Pepper-1D-1H-annotation-Table.pdf 2D spectra of pepper extract (MBBBI_18P08-LR_Pepper 2DNMR.zip) for complementary metabolite assignments	Text (.pdf) zip files (.zip, TopSpin Bruker format when unzipped)	recherche.data.gouv (https://doi.org/10.57745/U9K1BB) [9]
Data set 4	Eggplant-1D-1H-annotation-Table.pdf 2D spectra of eggplant extract (ACBBI_17P05-LR_Eggplant 2DNMR.zip) for complementary metabolite assignments	Text (.pdf) zip files (.zip, TopSpin Bruker format when unzipped)	recherche.data.gouv (https://doi.org/10.57745/AXXEXR) [10]
Data set 5	Pepper-2D-NMR-Unknown_1.pdf Pepper-2D-NMR-Unknown_2.pdf	Figures and Table (.pdf)	recherche.data.gouv (https://doi.org/10.57745/U9K1BB) [9]
Data file 6	Pepper_NMR_Metabolites.pdf	Text (.pdf)	ODAM (https://identifiers.org/odam.explorer:FR17PP009) [7]
Data file 7	Eggplant_NMR_Metabolites.pdf	Text (.pdf)	ODAM (https://identifiers.org/odam.explorer:FR17EP006) [8]
Data file 8	Pepper-1D-NMR-resonance-groups.pdf	Figures (.pdf)	recherche.data.gouv (https://doi.org/10.57745/U9K1BB) [9]
Data file 9	Eggplant-1D-NMR-resonance-groups.pdf	Figures (.pdf)	recherche.data.gouv (https://doi.org/10.57745/AXXEXR) [10]
Data file 10	Pepper NMR data: nmr_metabolites	Comma separated text file (.csv)	ODAM (https://identifiers.org/odam.explorer:FR17PP009) [7]
Data file 11	Eggplant NMR data: nmr_metabolites	Comma separated text file (.csv)	ODAM (https://identifiers.org/odam.explorer:FR17EP006) [8]
Data file 12	Pepper-PCA-Figure.pdf	Figures (.pdf)	ODAM (https://identifiers.org/odam.explorer:FR17PP009) [7]
Data file 13	Eggplant-PCA-Figure.pdf	Figures (.pdf)	ODAM (https://identifiers.org/odam.explorer:FR17EP006) [8]
Data file 14	Pepper-Eggplant-PCA-Figures.pdf	Figures (.pdf)	ODAM (https://identifiers.org/odam.explorer:FR17PP009 and https://identifiers.org/odam.explorer:FR17EP006) [7, 8]

recherche.data.gouv.fr/en [9, 10]) for 1D and 2D spectra, and an institutional data management system based on FAIR principles (ODAM, https://inrae.github.io/ODAM/, [17]) for pepper (https://pmb-bordeaux.fr/dataexplorer/?ds=FR17PP009 [7]) and eggplant data (https://pmb-bordeaux.fr/dataexplorer/?ds=FR17EP006 [8]).

Limitations

Due to its current low sensitivity, ^{1}H-NMR of polar extracts allowed the absolute quantification of major compounds, mostly primary metabolites but including major soluble sugars and organic acids crucial for fruit taste. Intermediaries of central metabolism such as sugar phosphates and

specialized metabolites such as glycoalkaloids, phenolics and isoprenoids should be determined, as relative or absolute contents, using dedicated protective solvent extraction [14] and complementary analytical strategies based on liquid chromatography coupled to mass spectrometry or to tandem mass spectrometry [2, 18].

Abbreviations

1D	Monodimensional
COSY	COrrelation SpectroscopY
DPA	Days post-anthesis
^{1}H-NMR	Proton nuclear magnetic resonance spectroscopy
HMBC	Heteronuclear Multiple Bond Correlation
HSQC	Heteronuclear Single Quantum Correlation
TOCSY	TOtal Correlation SpectroscopY
PCA	Principal component analysis

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Author contributions
Y.G. and A.M. designed the experiment. C.D., L.R., A.C. and M.B. performed the NMR analyses and metabolite quantifications. C.D., L.R., A.M. and D.J. curated the data. D.J. managed the data. AM wrote the initial version of the manuscript. AM, CD and DJ edited the manuscript. All authors read and approved the final version of the manuscript.

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Availability of data and materials
The spectral data described in this Data note can be freely and openly accessed on recherche.data.gouv repository under https://doi.org/10.57745/U9K1BB [9] and https://doi.org/10.57745/AXXEXR [10]. The processed data can be freely and openly accessed in ODAM data management system (https://identifiers.org/odam.explorer:FR17PP009 [7] and https://identifiers.org/odam.explorer:FR17EP006 [8]). Please see Table 1 for details and links to the data.

Declarations

Ethics approval and consent to participate
All local, national or international guidelines and legislation were adhered to in the production of this study.

Consent for publication
Not applicable.

Competing interests
The authors declare no competing interests.

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