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Data Article

Data from mass spectrometry, NMR spectra, GC–MS of fatty acid esters produced by *Lasiodiplodia theobromae*

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ABSTRACT

The data described herein is related to the article with the title "Fatty acid esters produced by *Lasiodiplodia theobromae* function as growth regulators in tobacco seedlings" C.C. Uranga, J. Beld, A. Mrse, I. Cordova-Guerrero, M.D. Burkart, R. Hernandez-Martinez (2016) [1]. Data includes nuclear magnetic resonance spectroscopy and GC–MS data used for the identification and characterization of fatty acid esters produced by *L. theobromae*. GC–MS traces are also shown for incubations in defined substrate, consisting in Vogel's salts supplemented with either 5% grapeseed oil or 5% glucose, the two combined, or 5% fructose. Traces for incubations in the combination of 5% grapeseed oil and 5% glucose for different fungal species are also included. Images of mycelium morphology when grown in 5% glucose with or without 5% grapeseed oil are shown due to the stark difference in mycelial pigmentation in the

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presence of triglycerides. High concentration gradient data for the plant model *Nicotiana tabacum* germinated in ethyl stearate (SAEE) and ethyl linoleate (LAEE) is included to show the transition between growth inhibition and growth induction in *N. tabacum* by these compounds.

Specifications Table

Subject area	Microbial biochemistry.
More specific subject area	Fatty acid metabolism by plant fungal pathogens.
Type of data	Mass spectrometry data, NMR spectra, GC–MS chromatograms, photography, microscopy, *N. tabacum* morphology and measurements of early growth in LAEE and SAEE concentration gradients.
How data was acquired	High resolution mass spectrometry: Agilent 6230 ESI-TOF MS. NMR: Varian 500 MHz instrument equipped with an XSens 2-channel NMR cold probe. GC–MS: Agilent 7890A GC system, connected to a 5975C VL MSD quadrupole MS (EI) mass spectroscopy. Olympus stereo microscope (SZX12).
Data format	Analyzed
Experimental factors	Material was purified with silica gel, HPLC and TLC for HR-MS and NMR analysis, followed by GC–MS. Carbon substrates were defined and simplified and subjected to further GC–MS.
Experimental features	Fungal samples were lyophilized, then extracted via a modified Folch extraction using 1:1 v/v dichloromethane and methanol along with 0.01% BHT as an antioxidant.
Data source location	University of California, San Diego, USA and CICESE, Ensenada, Mexico
Data accessibility	All relevant data is provided.

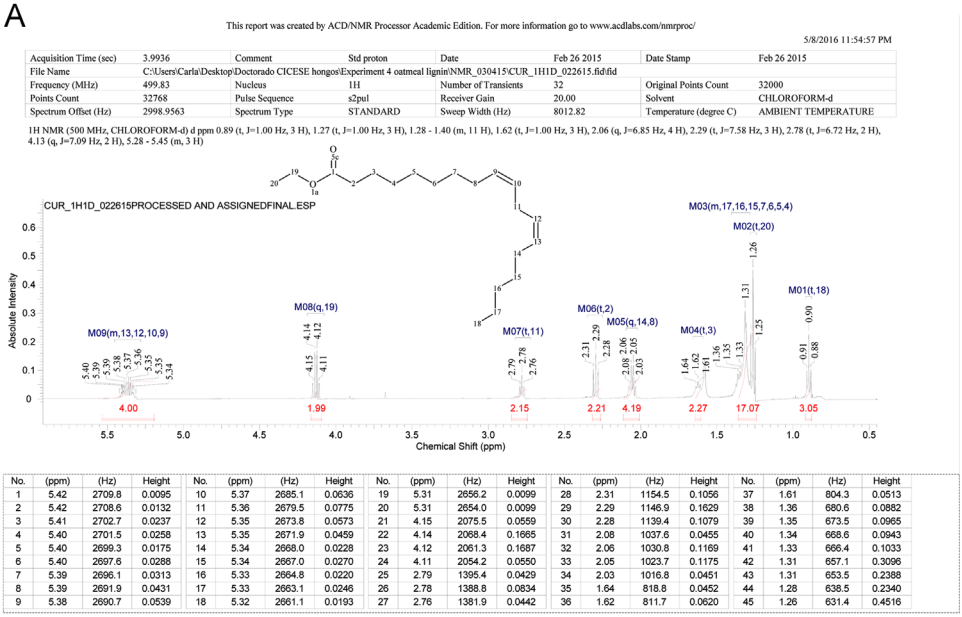
Value of the data

- This is the first report of fatty acid esters naturally produced by *Lasiodiplodia theobromae* and the other fungal species studied.
- Lipases from *L. theobromae* and *Neofusicoccum parvum* have broad substrate specificity that may be of interest for further characterization and potential biotechnological uses.
- Many of the fatty acid esters are novel for phytopathogenic fungi and might open exciting new research areas in fungal lipidomics and plant pathology.

1. Data

The data being shared consists in NMR spectra, as well as high-resolution mass spectrometry spectra and gas chromatography–mass spectrometry chromatograms used to identify fatty acid esters from the phytopathogenic fungus, *L. theobromae*. Other fungi such as *Neofusicoccum parvum, Fusarium oxysporum* f.sp. *lycopersici* and *Trichoderma asperellum* were also studied for comparison. Images of mycelial morphology for *L. theobromae* in different carbon sources are shown. Effects of fatty acid esters produced by *L. theobromae* in *N. tabacum* morphology are included. Concentration gradients for the most physiologically active compounds, ethyl stearate (SAEE) and ethyl linoleate (LAEE) are also shown.

A



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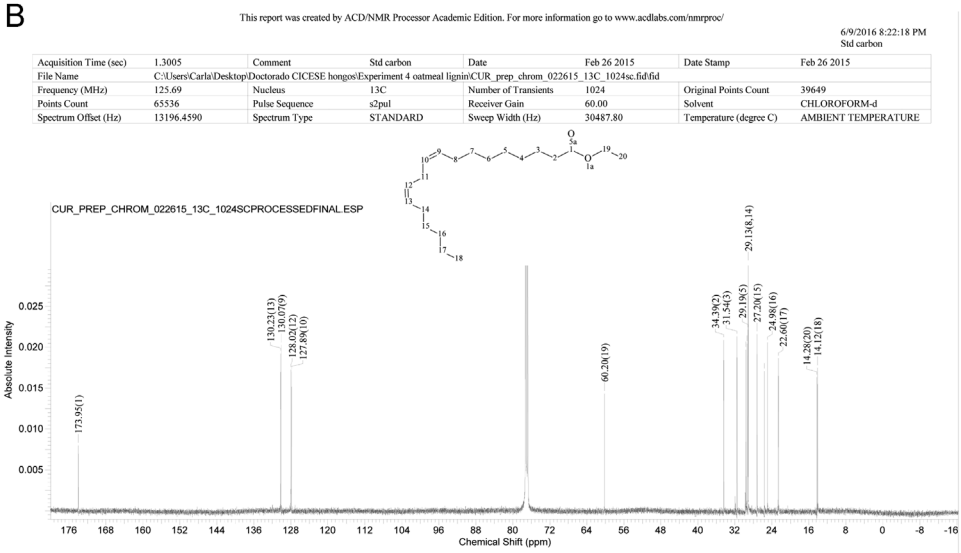
1	5.42	2709.8	0.0095	10	5.37	2685.1	0.0636	19	5.31	2656.2	0.0099	28	2.31	1154.5	0.1056	37	1.61	804.3	0.0513
2	5.42	2708.6	0.0132	11	5.36	2679.5	0.0775	20	5.31	2654.0	0.0099	29	2.29	1146.9	0.1629	38	1.36	680.6	0.0882
3	5.41	2702.7	0.0237	12	5.35	2673.8	0.0573	21	4.15	2075.5	0.0559	30	2.28	1139.4	0.1079	39	1.35	673.5	0.0965
4	5.40	2701.5	0.0258	13	5.35	2671.9	0.0459	22	4.14	2068.4	0.1665	31	2.08	1037.6	0.0455	40	1.34	668.6	0.0943
5	5.40	2699.3	0.0175	14	5.34	2668.0	0.0228	23	4.12	2061.3	0.1687	32	2.06	1030.8	0.1169	41	1.33	666.4	0.1033
6	5.40	2697.6	0.0288	15	5.34	2667.0	0.0270	24	4.11	2054.2	0.0550	33	2.05	1023.7	0.1175	42	1.31	657.1	0.3096
7	5.39	2696.1	0.0313	16	5.33	2664.8	0.0220	25	2.79	1395.4	0.0429	34	2.03	1016.8	0.0451	43	1.31	653.5	0.2388
8	5.39	2691.9	0.0431	17	5.33	2663.1	0.0246	26	2.78	1388.8	0.0834	35	1.64	818.8	0.0452	44	1.28	638.5	0.2340
9	5.38	2690.7	0.0539	18	5.32	2661.1	0.0193	27	2.76	1381.9	0.0442	36	1.62	811.7	0.0620	45	1.26	631.4	0.4516

B



No.	Atom	Exp. Shift (ppm)	No.	Atom	Exp. Shift (ppm)	No.	Atom	Exp. Shift (ppm)	No.	Atom	Exp. Shift (ppm)
1	20	14.28	6	15	27.20	11	10	127.89	16	5	29.19
2	19	60.20	7	14	29.13	12	9	130.07	17	4	29.61
3	18	14.12	8	13	130.23	13	8	29.13	18	3	31.54
4	17	22.60	9	12	128.02	14	7	27.21	19	2	34.39
5	16	24.98	10	11	29.36	15	6	25.63	20	1	173.95

Fig. 1. Analysis of the compound isolated from *Lasiodiplodia theobromae*. A: ^{1}H NMR spectrum. B: ^{13}C NMR spectrum identified as linoleic acid ethyl ester (LAEE).

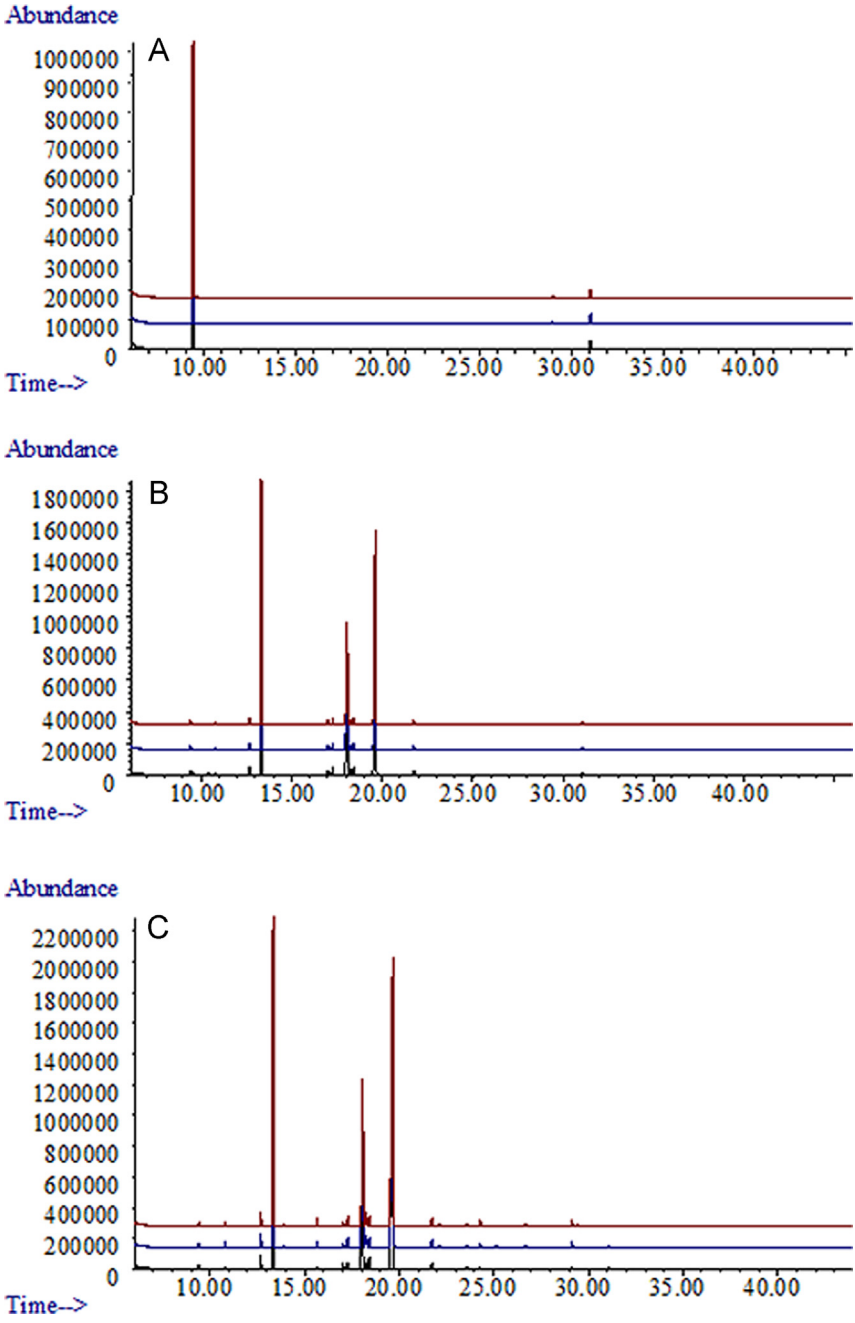


Fig. 2. Overview of GC/MS traces from *Lasiodiplodia theobromae* strains incubated in oatmeal. A: Three replicates of negative control (oatmeal only). B: Three replicates of strain UCD256Ma. C: Three replicates of strain MXL28.

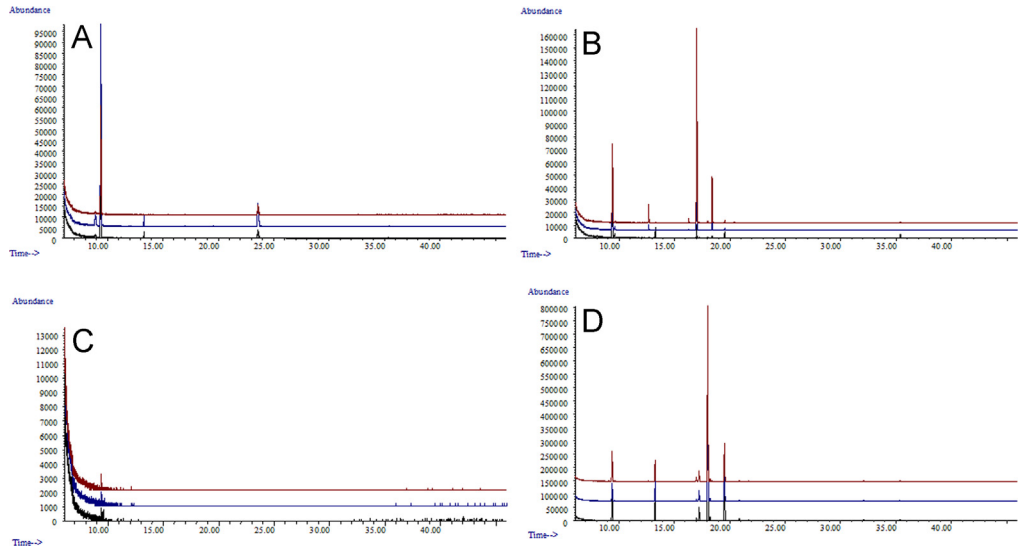


Fig. 3. GC/MS traces of *Lasiodiplodia theobromae* (UCD256Ma) incubated in Vogel's salts with A; 5% glucose. B; 5% grapeseed oil. C; 5% fructose. D; a combination of 5% glucose+5% grapeseed oil as carbon sources.

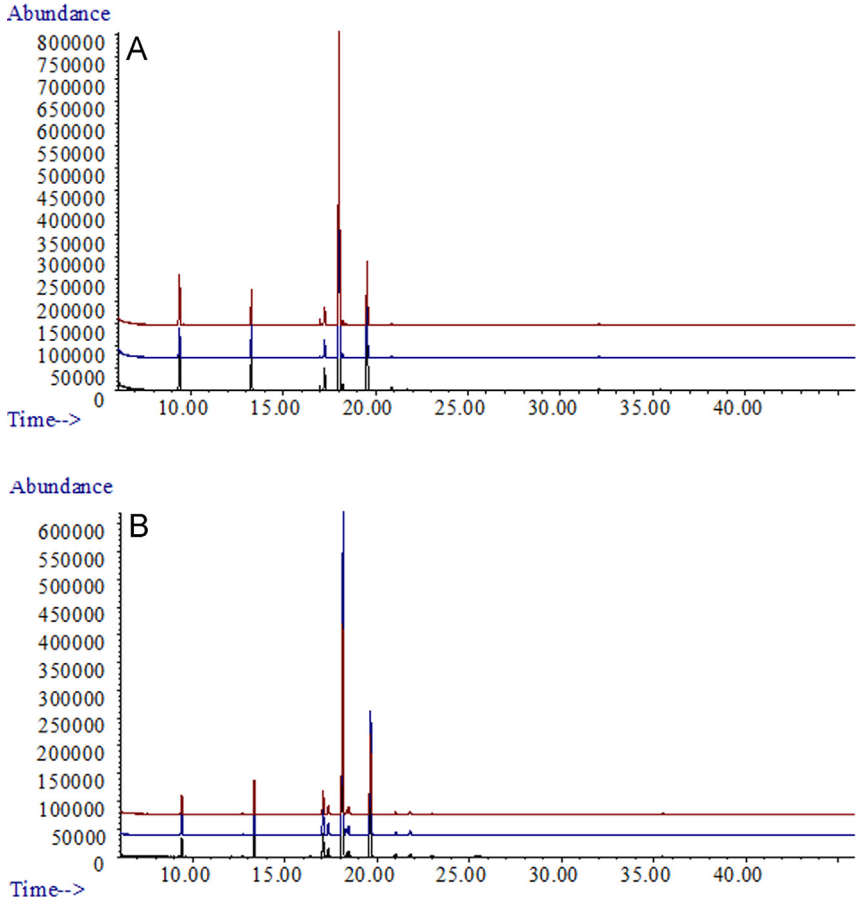


Fig. 4. GC/MS traces of A; *Lasiodiplodia theobromae* (UCD256Ma) and B; *Neofusicoccum parvum* (UCD646So). Both were grown in 5% glucose+5% grapeseed oil combined.

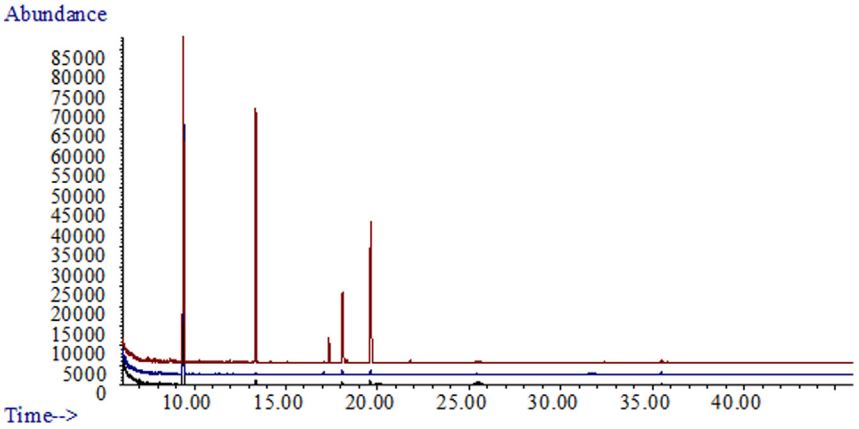


Fig. 5. GC/MS traces of a soil borne pathogen *Fusarium oxysporum* f. sp. *lycopersici* incubated in Vogel's salts with both 5% glucose and 5% grapeseed oil.

Table 1
Mean area under the curve (AUC) data presented as percent yields of total of the compounds identified in each strain and in each carbon source.

Identified compounds	5% grapeseed oil	5% glucose	5% grapeseed oil	5% glucose + 5% grapeseed oil			
	Grapeseed oil Fischer esterification (%)	*L. theobromae* UCD 256 Ma (%)	*L. theobromae* UCD 256 Ma (%)	*L. theobromae* UCD 256 Ma (%)	*N. parvum UCD646So* (%)	*F. oxysporum* (%)	*T. asperellum* (%)
Methyl hexadecanoate	1.9	0.0	2.7	0.0	0.0	0.0	0.0
Ethyl hexadecanoate (PAEE)	2.5	16.8	1.6	6.1	6.0	44.8	2.8
Hexadecanoate, 2-methylpropyl ester	0.0	0.0	0.0	0.0	0.0	0.0	0.0
9-Octadecenoate (Z)- methyl ester	31.2	0.0	44.0	0.7	5.2	0.0	20.9
Octadecanoate ethyl ester (SAEE)	1.0	0.0	0.2	3.9	2.1	4.9	0.0
9-Octadecenoate (Z), ethyl ester (OAEE)	41.9	0.0	0.0	70.4	59.9	15.3	69.5
9-Octadecenoate (E) ethyl ester	0.3	0.0	0.0	0.9	0.9	0.0	0.0
9,12-Octadecadienoate (Z,Z)-, methyl ester	9.2	0.0	9.4	0.1	1.5	0.0	0.0
9,12-Octadecadienoate (Z,Z) ethyl ester (LAEE)	11.7	0.0	40.9	16.2	23.7	35.0	6.8
9,12,15-Octadecatrienoate (Z,Z,Z)-ethyl ester)	0.2	0.0	1.2	1.7	0.7	0.0	0.0
2H-1-Benzopyran, 3,4-dihydro- (R ± mellein)	0.0	83.2	0.0	0.0	0.0	0.0	0.0

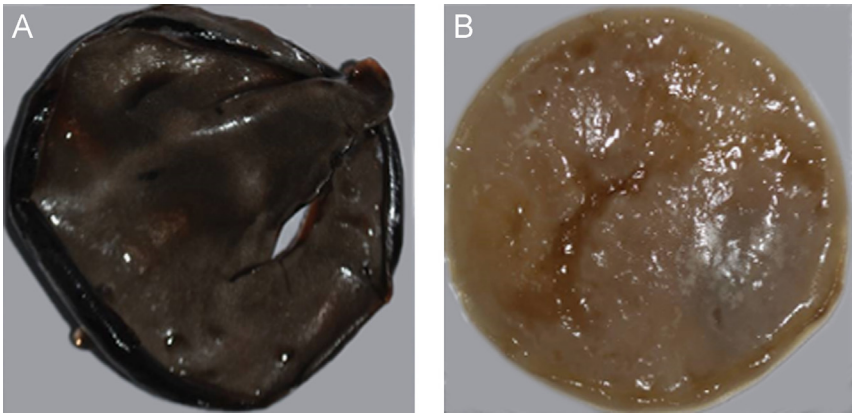


Fig. 6. Morphology of *Lasiodiplodia theobromae* (UCD256Ma). A: *Lasiodiplodia theobromae* incubated in 5% glucose in Vogel's salts. B: *Lasiodiplodia theobromae* incubated in 5% glucose and 5% grapeseed oil in Vogel's salts.

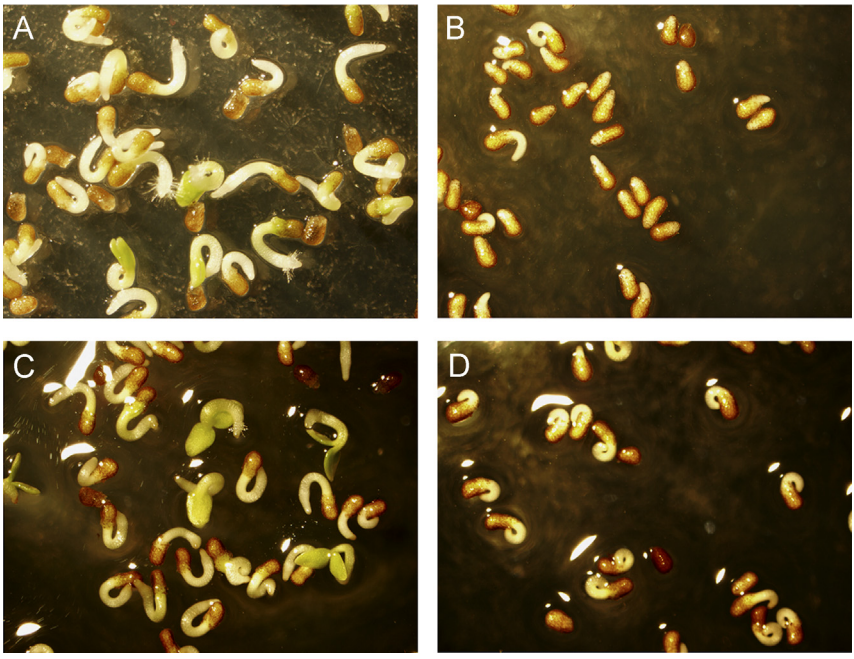


Fig. 7. Seed germination in *Nicotiana tabacum* exposed to 100 μg/mL free palmitate, palmitate ethyl ester or linoleate ethyl ester emulsified with 0.08% kolliphor P-188 in Murashige and Skoog salts with Gamborg vitamins, supplemented with 3% sucrose, and 0.4% PPM. A: negative control. B: 0.1 mg/mL LAEE. C: 0.1 mg/mL PA. D: 0.1 mg/mL PAEE.

2. Experimental design, materials and methods

For purification, mass spectrometry and nuclear magnetic resonance (NMR) [2,3], a modified Folch extraction [4] was standardized as described in [1], (Supplementary Data Set A and Fig. 1A, B). Carbon source effects were then studied in *L. theobromae* using the standardized Folch extraction. Fatty acid ester production was studied in the other fungal species *N. parvum, F. oxysporum* and *T. asperellum* for comparison. All samples, including the positive controls, were analyzed for naturally produced fatty

Fig. 8. Morphology of *Nicotiana tabacum* germinated in FAE and grown 45 days in Murashige–Skoog+3% sucrose. A; three biological replicates of *N. tabacum* without FAE. B; 0.2 mg/mL PAEE. C; 0.2 mg/mL LAEE. D; 0.2 mg/mL SAEE. E; 3.13 μg/mL SAEE. F; 0.2 mg/mL crude extract of *Lasiodiplodia theobromae* incubated in 5% grapeseed oil+5% glucose.

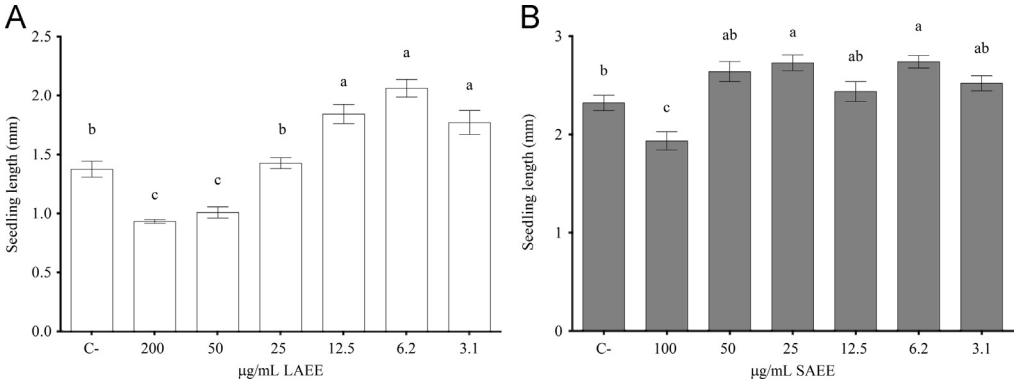


Fig. 9. High concentration ranges of ethyl linoleate (LAEE) and ethyl stearate (SAEE) in *Nicotiana tabacum* seedling germination, showing a concentration dependent transition from growth inhibition to growth induction in each compound.

acid ethyl esters by gas chromatography/mass spectrometry (GC–MS) as described in [1] (Figs. 2–5). The data was expressed as percent yield of each compound from the total compounds identified (Table 1). Morphology of *L. theobromae* incubated in 5% glucose was documented by photography and compared to the morphology in 5% glucose+5% grapeseed oil (Fig. 6). With the aim to test the effect of the isolated compounds *in planta*, we chose tobacco (*Nicotiana tabacum),* a well-studied plant model [5] to measure growth as described in [1]. The length of the seedling was measured after 7–10 days post-sowing using calibrated Image J software [6] from cotyledon tip to root tip for each experimental condition. Morphology was also assessed and documented 45 days post-dosing and sowing (Figs. 7–8). A high concentration gradient for the most physiologically active fatty acid esters found and described in [1] was performed by germinating the plant model *N. tabacum* in SAEE from 100 to 3.1 μg/mL and LAEE from 200 to 3.1 μg/mL (Fig. 9).

Acknowledgments

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Appendix A. Supplementary material

Supplementary data associated with this article can be found in the online version at http://dx.doi.org/10.1016/j.dib.2016.05.003.

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