Data Article

Mass spectrometry data from a quantitative analysis of protein expression in gills of immuno-challenged blue mussels (*Mytilus edulis*)

K. Hörnaeus [a], J. Guillemant [a], J. Mi [a], B. Hernroth [b,c], J. Bergquist [a], S. Bergström Lind [a,*]

[a] *Department of Chemistry – BMC, Analytical Chemistry and SciLifeLab, Uppsala University, Box 599, SE – 75124 Uppsala, Sweden*
[b] *The Royal Swedish Academy of Sciences, Sven Lovén Center for Marine Science, Kristineberg 566, SE – 451 78 Fiskebäckskil, Sweden*
[c] *Department of Natural Science, Kristianstad University, SE – 291 88 Kristianstad, Sweden*

A R T I C L E   I N F O

A B S T R A C T

Here, we provide the dataset associated with our research article on the potential effects of ocean acidification on antimicrobial peptide (AMP) activity in the gills of *Mytilus edulis*, "Impact of ocean acidification on antimicrobial activity in gills of the blue mussel (*Mytilus edulis*)" [1]. Blue mussels were stimulated with lipopolysaccharides and samples were collected at different time points post injection. Protein extracts were prepared from the gills, digested using trypsin and a full in-depth proteome investigation was performed using liquid chromatography coupled to tandem mass spectrometry (LC–MS/MS). Protein identification and quantification was performed using the MaxQuant 1.5.1.2 software, "MaxQuant enables high peptide identification rates, individualized p.p.b.-range mass accuracies and proteome-wide protein quantification" [2].
© 2016 The Authors. Published by Elsevier Inc. This is an open access article under the CC BY license (http://creativecommons.org/licenses/by/4.0/).

Specifications Table

Subject area	*Biology*
More specific subject area	*Protein expression in Mytilus edulis*
Type of data	*Table,.xlsx file*
How data was acquired	*Data dependent LC-MS/MS acquired on a Q Exactive instrument (Thermo Fisher Scientific) coupled to a ultra nanoflow high-performance liquid chromatography (HPLC) system (EASY-nLC™ 1000, Thermo Fisher Scientific)*
Data format	*Processed, analyzed*
Experimental factors	a) *Lipopolysaccharide stimulation of blue mussels and protein extraction from gills* a) *LC-MS/MS analysis for protein identification and quantification*
Experimental features	*Blue mussels were stimulated with lipopolysaccharides and samples were collected at different time points post injection. Protein extracts were prepared from the gills, digested using trypsin and a full in-depth proteome investigation was performed using LC-Orbitrap MS/MS technique. The spectra (.RAW) were acquired using Xcalibur software 3.0.63 and further database searches were performed using MaxQuant 1.5.1.2. The search results were stored as.xls-files.*
Data source location	*Uppsala, Sweden*
Data accessibility	*Data are within this article (Supplementary Table 1)*

Value of the data

- The data further validate the protein expression changes presented in Hernroth et al. [1].
- The data can be used to validate protein identification in *Mytilus edulis* from other studies.
- The in depth proteomic data enables comparison with RNA expression data.

1. Data

This dataset comprise the output file (Supplementary Table 1, available online) from the database search of LC–MS/MS raw files obtained from bottom-up MS analysis of gills from *Mytilus edulis* immune-challenged by lipopolysaccharide injection. Samples were collected at five time points post injection of lipopolysaccharide (Table 1). One control group of mussels injected with only *Mytilus* physiological saline (PS)-buffer was included. Each group included five individual mussels.

2. Experimental design, materials and methods

2.1. Experimental set up

Thirty mussels were kept in the running seawater system of SLC-Kristineberg (∼32 PSU, 14 °C) and divided into six 15 L basins with five individuals in each. Bacterial contamination was avoided by pre-challenging with lipopolysaccharide (LPS; #L7261, Sigma Aldrich) dissolved in PS-buffer [3]. One control group of mussels was injected with only PS-buffer and the other five groups were injected with 0.2 μg LPS g^{-1} mussel (wet weight), into the adductor muscle. The gills of mussels from the control group were dissected at time 0 followed by dissection of one group at a time after 0.5, 1.5, 3, 5 and 8 h post injection. The dissected gill tissues were immediately put on dry ice before being stored at −80 °C until further prepared.

Table 1.
Samples included for each time point post injection.

Sample number	Time post injection (h)
1–5	Ctrl
6–10	0.5
11–15	1.5
16–20	3
21–25	5
26–30	8

2.2. Sample preparation

The samples were homogenized in 9 M urea, 20 mM HEPES using a micro pestle. Proteins were extracted by sonication using a probe with a 3 mm tip (10×1 s, amplitude 30%) followed by centrifugation at 16,000g for 20 min at 4 °C. The total protein concentration in the samples was analyzed using Coomassie assay, with bovine serum albumin (BSA) as standard [4]. Aliquots corresponding to 20 µg protein were reduced with dithiothreitol (DTT) and alkylated with iodoacetamide (IAA). After four times dilution with 50 mM ammonium bicarbonate, trypsin was added in a trypsin:protein ratio of 1:20 and digestion was performed overnight. Thereafter the samples were purified by Pierce C18 Spin Columns (Thermo Scientific), dried and resolved in 60 µL 0.1% formic acid.

2.3. LC–MS/MS

The samples were analyzed using a QExactive Plus Orbitrap mass spectrometer (Thermo Fisher Scientific, Bremen, Germany) equipped with a nano electrospray ion source. The peptides were separated by reversed phase liquid chromatography using an EASY-nLC 1000 system (Thermo Fisher Scientific). A set-up of pre-column and analytical column was used. The pre-column was a 2 cm EASY-column (1D 100 µm, 5 µm C18) (Thermo Fisher Scientific) while the analytical column was a 10 cm EASY-column (ID 75 µm, 3 µm, C18; Thermo Fisher Scientific). Peptides were eluted with a 90 min linear gradient from 4% to 100% acetonitrile at 250 nL min^{-1}. The mass spectrometer was operated in positive ion mode acquiring a survey mass spectrum with resolving power 70,000 (full width half maximum), m/z = 400-1750 using an automatic gain control (AGC) target of 3×10^6. The 10 most intense ions were selected for higher-energy collisional dissociation (HCD) fragmentation (25% normalized collision energy) and MS/MS spectra were generated with an AGC target of 5×10^5 at a resolution of 17,500. The mass spectrometer worked in data-dependent mode.

2.4. Mass spectrometry data handling

The acquired data (.RAW-files) were processed by MaxQuant 1.5.1.2 [2] and database searches were performed using the implemented Andromeda search engine. MS/MS spectra were correlated to a FASTA database containing proteins from *Mytilus* extracted from the NCBI database (release June 2015). A decoy search database, including common contaminants and a reverse database, was used to estimate the identification false discovery rate (FDR). An FDR of 1% was accepted. The search parameters included: maximum 10 ppm and 0.6 Da error tolerances for the survey scan and MS/MS analysis, respectively; enzyme specificity was trypsin; maximum one missed cleavage site allowed; cysteine carbamidomethylation was set as static modification and oxidation (M) was set as variable modification. For protein identification, only peptides with a minimum of seven amino acids and at least one unique peptide were accepted.

Acknowledgments

Transparency document. Supplementary material

Transparency document associated with this article can be found in the online version at http://dx. doi.org/10.1016/j.dib.2016.05.073.

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.2016.05.073.

References

[1] B. Hernroth, S. Baden, H. Tassidis, K. Hörnaeus, J. Guillemant, S. Bergström Lind, J. Bergquist, Impact of ocean acidification on antimicrobial activity in gills of the blue mussel (*Mytilus edulis*), Fish Shellfish Immunol. (2016), in press, 10.1016/j.fsi.2016.04.007.
[2] J. Cox, M. Mann, MaxQuant enables high peptide identification rates, individualized p.p.b.-range mass accuracies and proteome-wide protein quantification, Nat. Biotech. 26 (2008) 1367–1372. http://dx.doi.org/10.1038/nbt.1511.
[3] B. Hernroth, The influence of temperature and dose on antimicrobial peptide response against lipopolysaccharide in the blue mussel, *Mytilus edulis*, Fish Shellfish Immunol. 14 (2003) 25–37.
[4] M.M. Bradford, A rapid and sensitive method for the quantitation of microgram quantities of protein utilizing the principle of protein-dye binding, Anal. Biochem. 72 (1976) 248–254.