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Data Article

Peptidomic data in porcine duodenal effluents after oral administration of micellar casein

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ABSTRACT

The data in this article are related to the research publication "Digestion of micellar casein in duodenum cannulated pigs. Correlation between in vitro simulated gastric digestion and in vivo data" (Miralles et al., Food Chemistry, 2021, 343, 128428). Pig duodenum effluents were collected with a T-shaped cannula 15 min before and during digestion over 150 min after casein intake. The casein degradation profile of individual pigs during digestion is presented. All identified peptide sequences at different digestion times for six subjects are provided. The peptide profile of digests in the form of heat maps is shown for α_{s1}-, α_{s2}-, β- and κ-casein. The sum of amino acids belonging to peptides released from β- and α_{s1}-casein has been used to determine correlation coefficients and range the inter-individual variability. Finally, the global amino acid composition, isoelectric point and sequence length of all released peptides has been determined.

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Specifications Table

Subject	Food Science
More specific subject area	Proteomics and biochemistry
Type of data	Table, Figures
How data was acquired	PAGE-SDS + imaging (VersaDoc™ MP 5000 system, Bio-Rad Laboratories) RP-HPLC-MS/MS (Ion-trap Esquire 3000 (Bruker Daltonics)
Data format	Raw and analyzed
Parameters for data collection	Six Large White × Landrace × Piétrain adults pigs of about 40 kg live weight were fitted with a T-shaped cannula surgically placed 10 cm downstream from the pylorus to collect duodenum effluents 15 min before and 5, 10, 15, 20, 30, 45, 60, 90, 120 and 150 min after ingestion of micellar casein.
Description of data collection	LC-MS/MS files were processed with Data analysis and Biotools (Bruker Daltonics). Sequencing was performed with Mascot. The frequency of appearance of each amino acid identified as part of a peptide sequence was used to build heat maps. Spearman correlation coefficient calculated with the frequency of appearance of each amino acid was used to show the inter-individual variability. In the identified peptides the amino acids composition was calculated by using ProtParam tool (Expasy) and isoelectric point and number of amino acids were ranged using Peptide Analyzer (Thermofisher Scientific).
Data source location	Instituto de Investigación en Ciencias de la Alimentación CIAL (CSIC-UAM) Madrid, Spain
Data accessibility	Mendeley Data https://doi.org/10.17632/5vn48f492w.2
Related research article	B. Miralles, J. Sanchón, L. Sánchez-Rivera D. Martínez-Maqueda Y. Le Gouar, D. Dupont, L. Amigo, I. Recio. Digestion of micellar casein in duodenum cannulated pigs. Correlation between in vitro simulated gastric digestion and in vivo data. Food Chem https://doi.org/10.1016/j.foodchem.2020.128424

Value of the Data

- The duodenal contents in the pig can be representative of micellar casein digestion in humans
- Peptide sequences released in the upper intestinal tract, which may have physiological implications, are available for future assays
- These data illustrate the inter-individual variability of the peptide profile in duodenal contents after casein digestion.
- These *in vivo* data are useful to validate static and dynamic gastrointestinal simulations in terms of protein degradation and peptide formation

1. Data Description

Fig. 1 shows the SDS-PAGE protein profiles at duodenum during pig digestion of casein in three different individuals. Intact caseins, whey proteins, degradation products thereof and digestive enzymes were identified by in-gel digestion and MALDI-MS/MS in the electrophoretic profile from one of the subjects [1].

Peptide sequencing was conducted by HPLC-MS/MS by matching with the main genetic variants from milk caseins. All peptide sequences identified in the duodenal content of each individual and time of sample collection are listed in Mendeley (https://data.mendeley.com/datasets/5vn48f492w/1). In total, 3895 peptides were sequenced, 1221 from α_{s1}-casein, 346 from α_{s2}-casein, 1489 from β-casein and 784 from κ-casein.

Heat maps built with the frequency of appearance of each amino acid identified as part of a peptide sequence from α_{s1}-, α_{s2}-, β- and κ-casein, after 5, 10, 15, 20, 30 45, 60, 90, 120, and 150 min of micellar casein administration are shown in Figs. 2–5, respectively. The green color represents low frequency and red high frequency. For α_{s1}-casein and β-casein, an important number of peptides were found at the C-terminal region of the protein. In addition,

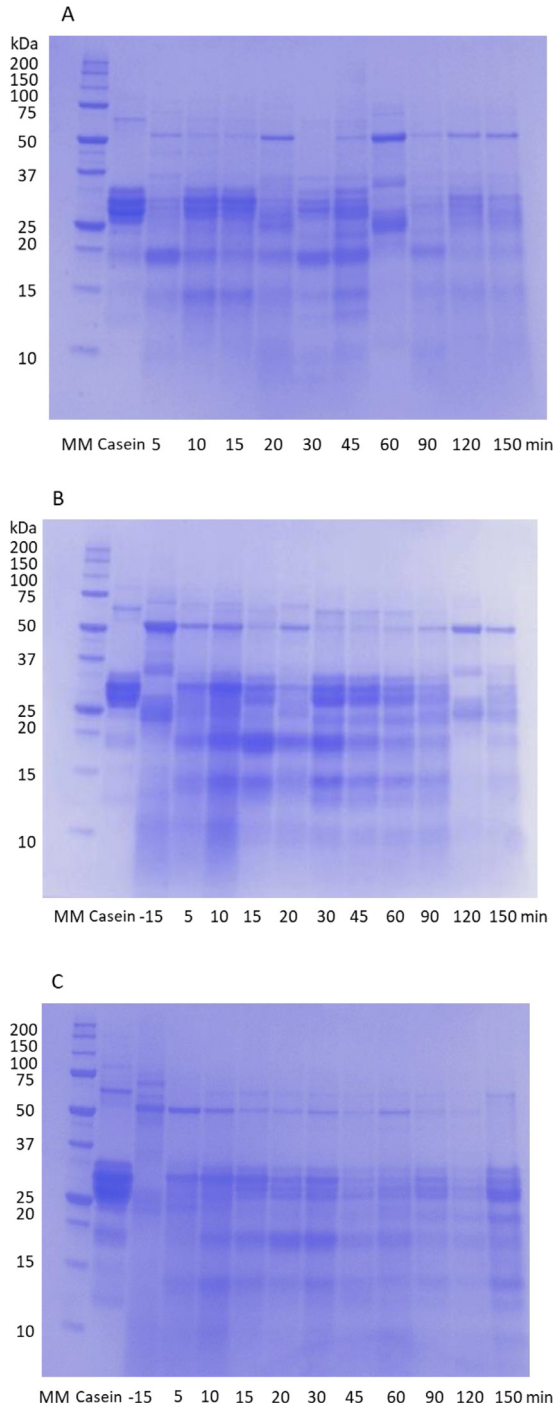

Fig. 1. SDS-PAGE of duodenal micellar casein digests from three individual animals. Each lane corresponds to micellar casein or a collected duodenal sample (A) from 5 to 150 min, (B, C) from 15 min before casein ingestion to 150 min.

Fig. 2. Heat map built with the frequency of appearance of each amino acid identified as part of a peptide sequence of α_{s1}-casein (199 amino acid residues), after 5, 10, 15, 20, 30 45, 60, 90, 120, and 150 min of micellar casein administration. The green color represents low frequency and red high frequency. Each line represents a subject (n=6).

Fig. 3. Heat map built with the frequency of appearance of each amino acid identified as part of a peptide sequence from α_{s2}-casein (207 amino acid residues), after 5, 10, 15, 20, 30 45, 60, 90, 120, and 150 min of micellar casein administration. The green color represents low frequency and red high frequency. Each line represents a subject (n=6).

Fig. 4. Heat map built with the frequency of appearance of each amino acid identified as part of a peptide sequence from β-casein (209 amino acid residues), after 5, 10, 15, 20, 30 45, 60, 90, 120, and 150 min of micellar casein administration. The green color represents low frequency and red high frequency. Each line represents a subject (n=6).

Fig. 5. Heat map built with the frequency of appearance of each amino acid identified as part of a peptide sequence from κ-casein (169 amino acid residues), after 5, 10, 15, 20, 30 45, 60, 90, 120, and 150 min of micellar casein administration. The green color represents low frequency and red high frequency. Each line represents a subject (n=6).

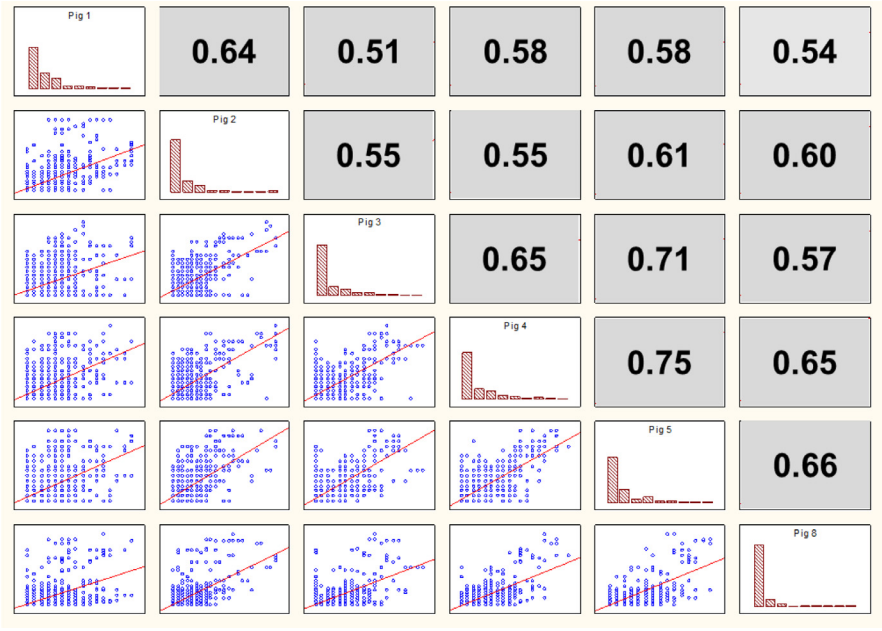

Fig. 6. Interindividual variability (Spearman correlation coefficient) calculated with the frequency of appearance of each amino acid identified as part of an identified peptide sequence after 5, 10, 15, 20, 30 45, 60, 90, 120, and 150 min of micellar casein administration (n=6).

peptides from regions 24-40 from α_{s1}-casein were also abundant in duodenal effluents at all-time points. For α_{s2}- and κ-casein, those regions with the largest number of identified peptides are around residues 85-90 and 110-120 from α_{s2}-casein, and residues 40 to 50 from κ-casein. Inter-individual variability (Spearman correlation coefficient) calculated with the frequency of appearance of each amino acid identified as part of an identified peptide sequence after micellar casein administration is shown in Fig. 6.

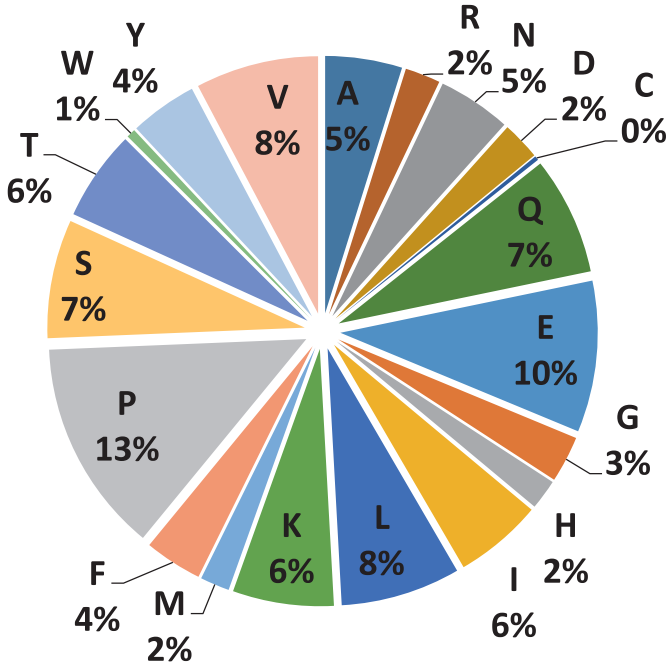

Fig. 7. Amino acid (one letter code) composition of the identified peptides in duodenal effluents from 6 individuals after 5, 10, 15, 20, 30 45, 60, 90, 120, and 150 min of micellar casein administration.

The amino acid composition in the identified peptides in duodenal effluents from 6 individuals after 5 to 150 min of micellar casein administration is shown in Fig 7. A high occurrence of proline and glutamic acid (13 and 10%, respectively) is followed by valine and leucine, with average relative shares of 8% each.

Identified sequences distributed by isoelectric point range are shown in Fig 8. The highest percentage of peptides, 28%, showed isoelectric points in the range from 3 to 4. The rest are widely distributed with relative shares from 3 to 14% at all isoelectric point ranges except range 8 to 9 where less than 1% of peptides are comprised.

Fig 9 shows the stratified bar graph with percentage of identified peptides by number of amino acid residues. The group of peptides with a number of amino acid residues between 6 and 10 is the most important, with relative shares between 12 and 16%, while percentages lower than 3.5% were observed for the rest.

2. Experimental Design, Materials and Methods

2.1. Animal experiments

The animals were male pigs Large White × Landrace × Piétrain of ca. 40 kg live weight. Two weeks before starting the trials, 10 animals were surgically fitted with a T-shaped cannula in the duodenum (10 cm downstream from the pylorus). After surgery, the animals were housed within a ventilated room with controlled temperature (21 °C) in individual slatted pens. During convalescence and over the period of the trial, excluding sampling days, pigs received a diet appropriate for their age and physiological state, which consists of 800 g/d of a pig feed concentrate (Cooperl Arc Atlantique®, Pelstan, France) containing 16% proteins, 1% fat, 4% cellulose and 5% mineral matter. Furthermore, they had free access to water.

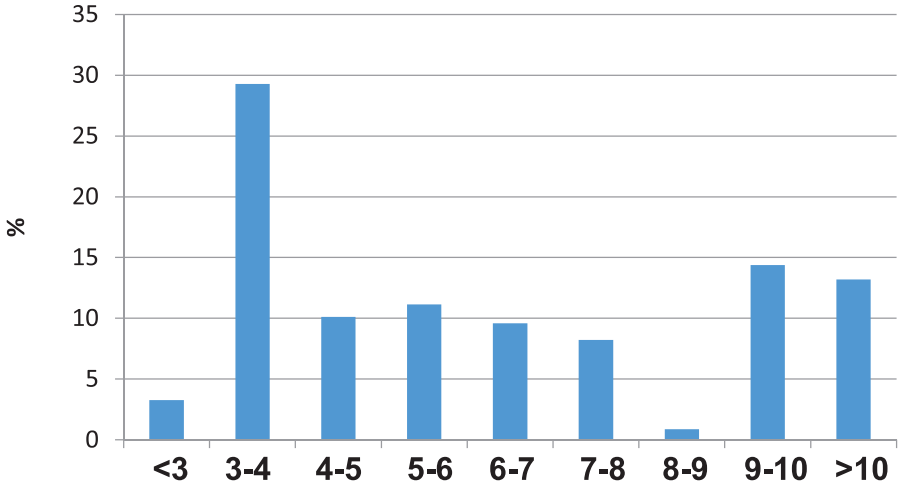

Fig. 8. Percentage of identified peptides within each isoelectric point range. Identified peptides in duodenal effluents from 6 individuals after 5, 10, 15, 20, 30 45, 60, 90, 120, and 150 min of micellar casein administration.

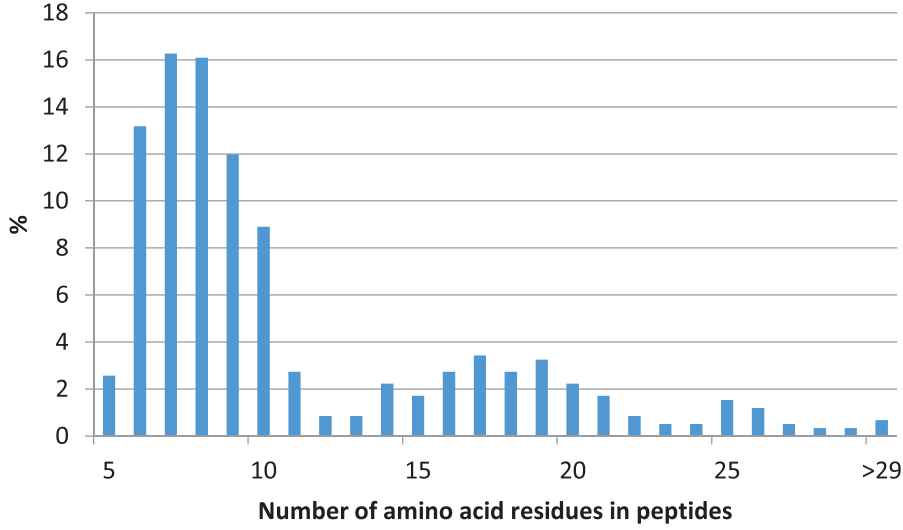

Fig. 9. Percentage of identified peptides by number of amino acid residues. Identified peptides in duodenal effluents from 6 individuals after 5, 10, 15, 20, 30 45, 60, 90, 120, and 150 min of micellar casein administration.

The trial period lasted two weeks, whereby the sampling phases were separated by at least 2 days. Test meal (casein powder reconstituted in water 5% (w/v) adequate in quantity to cover the daily requirements of protein) was provided to the pigs for 10 min. Chromium-EDTA (110.8 ppm, w/w) had been previously added to the casein preparation as marker of the liquid phase. The animals had no access to water neither 1h before nor 7h after the meal delivery. Duodenum effluents were collected 15 min before and 5, 10, 15, 20, 30, 45, 60, 90, 120 and 150 min after ingestion of test meal in plastic bottles. The sampling was ended when 40ml were pooled or after 3 min of sampling time. Pefabloc®, (Sigma-Aldrich, St. Louis, MO, USA) at 2.5 mM final

concentration was added to the collecting containers. The digestive contents were subsequently weighed and freeze-dried.

2.2. Samples and reagents

Commercial micellar casein (77.84% protein measured by Kjeldahl) was from Ingredia S.A, Arras, France (Prodiet 85B, batch no. 141179). Unless otherwise specified, all chemicals were from Sigma-Aldrich (St. Louis, MO, USA).

2.3. Analysis of porcine duodenal effluents

2.3.1. Protein characterization by SDS-PAGE

SDS-PAGE was performed as previously described [2] but protein load of digestive contents was adjusted to 1 mg of protein/mL. Undigested casein was dissolved at 0.8 mg of protein/mL.

2.3.2. Peptide identification

Freeze-dried digests were reconstituted in solvent A (water:formic acid, 100:0.1, v/v) and centrifuged at 13000 × *g*, 10 min. 50 µL of supernatant was injected for each sample. Besides, an enriched phosphopeptide fraction from the samples was prepared by selective precipitation with addition of calcium chloride (1% w/v) and ethanol (50% v/v) [2].

HPLC-MS/MS was carried out using an Agilent 1100 HPLC system (Agilent Technologies, Waldbron, Germany), with a Mediterranea Sea C_{18} column (150 × 2.1 mm, Teknokroma, Barcelona, Spain) connected to an Esquire 3000 linear ion trap mass spectrometer (Bruker Daltonics GmbH, Bremen, Germany) fitted with an electrospray ionization source. For peptide elution, the flow was set at 0.2 mL/min and a linear gradient over 120 min from 0 to 45% of solvent B (acetonitrile:formic acid, 100:0.1, v/v) was employed. The mass/charge (*m/z*) ranged from 100 to 1200 and the target mass was set at 900 m/z. Data were processed by using Data Analysis (version 4.0, Bruker DaltonicsGmbH, Bremen, Germany). Peptide sequencing was performed using the MASCOT v2.4 protein search engine (Matrix Science) as previously described [3]. Regardless of its *P* value, a manual revision was done for each identification peptide spectrum.

2.3.3. Peptidomic analysis

The frequency of appearance of each amino acid identified as part of a peptide sequence was used to build heat maps with alignment in each parental protein (β-, α_{s1}-, α_{s2}, and κ-casein). Spearman correlation coefficient calculated with the frequency of appearance of each amino acid was used to determine the inter-individual variability. The amino acids composition of the identified peptides was calculated by using ProtParam tool (Expasy) and isoelectric point and number of amino acids were ranged using Peptide Analyzer (Thermofisher Scientific).

2.4. Statistical analysis

Spearman correlation matrices were built on the basis of frequency of appearance of amino acids included in the identified peptides. For the calculations, Statistica software (StatSoft Incl., Tulsa, OK, USA) was used.

Ethics Statement

All procedures were in accordance with the European Community guidelines for the use of laboratory animals (L358-86/609/EEC). The study was approved by the local committee for ethics in animal experimentation.

Credit Author Statement

B. Miralles: Methodology, Supervision, Writing-Original Draft; **J. Sanchón:** Investigation, Formal analysis; **L. Sánchez-Rivera:** Investigation, Formal analysis; **D. Martínez-Maqueda:** Investigation, Formal analysis; **Y. Le Gouar:** Investigation, **D. Dupont:** Conceptualization, Resources; **L. Amigo:** Writing-Review & Editing, **I. Recio**: Conceptualization, Supervision, Writing-Review & Editing

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships which have, or could be perceived to have influenced the work reported in this article.

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