SEPARATAS STUDY OF THE MICRO AND MACRO ELEMENTS DISTRIBUTION IN COW MILK SAMPLES BY INDUCTIVELY COUPLED PLASMA OPTICAL EMISSION SPECTROMETRY

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The milk is a complex fluid that has all nutrients required for the growth and development of the newborn. All kind of milk contain specific proteins, fats designed to be easily digested, minerals, vitamins and others components which may have important roles. The lipids are present in the milk as emulsified globules coated with a membrane and the proteins in form of micelles in a colloidal dispersion. Most of minerals and all lactose are present free in aqueous solution. This milk compartimentation influences the mineral availability as nutritive or nonnutritive agent. Several appropriated digestion or solubilization methods have been proposed for determination of inorganic species present in the whole milk. However, to increase the knowledge about the physiological activity of the milk minerals, the total amount determination is not enough. The establishment of the distinct chemical interactions among milk components has also to be appraised. In this work the protein precipitation with trichloroacetic acid (TCA) was compared with proteolytic enzymes (papain and pepsin) to evaluate the Fe, Zn, Ca and Mg interactions regarding aqueous and protein milk phases. The TCA reagent precipitated the milk protein in its isoeletric point. The enzymes denatured the milk proteins cleaving their peptide bounds. The two phases produced were easily centrifuged being the supernatant collected for further mineral quantification. A closed microwave-oven digestion was performed to appraise the total inorganic amount in the whole milk. The determinations were carried out by inductively coupled plasma optical emission spectrometry (ICP-OES) with axial view and CCD detector. The results are showed in the Table 1.

| Table 1. | Results | obtained | for a milk | sample | digested | in a | closed | micro | wave-o | ven a | ind | treated | with |
|----------|---------|------------|------------|-----------|----------|------|--------|-------|--------|-------|-----|---------|------|
| TCA 10 | % w/v a | ind pepsii | n 5.0 % w/ | v solutio | ons. | | | | | | | | |

| Sample | Ca (mg l ⁻¹) | | | Fe (μ g l ⁻¹) | | | $Mg (mg l^{-1})$ | | | Zn (mg l ⁻¹) | | |
|-----------|--------------------------|-------------------|-------|--------------------------------|------|------|------------------|-----|------|--------------------------|------------|------------|
| treatment | | | | | | | | | | | | |
| | | Milk ^a | | Milk | | | Milk | | | Milk | | |
| | 1 | 2 | 3 | 1 | 2 | 3 | 1 | 2 | 3 | 1 | 2 | 3 |
| Acid | 1188 | 1579 | 1401 | 14800 | 258 | 154 | 106 | 113 | 125 | 7.20 | 4.00 | 5.43 |
| digestion | $\pm .7^{b}$ | ± 48 | ± 28 | ± 100 | ±24 | ± 56 | ± 1 | ± 4 | ± 2 | ± 0.05 | ± 0.11 | ± 0.24 |
| TCA | 1225 | 1723 | 1460 | 435 ± | < DL | < DL | 107 | 121 | 131 | 7.75 | 4.58 | 5.45 |
| 10 % w/v | ± 54 | ± 35 | ± 113 | 33 | | | ± 5 | ± 4 | ± 10 | ± 0.36 | ± 0.07 | ± 0.94 |
| PEP | 1051 | 1453 | 1213 | 11700 | 328 | 306 | 93.1 | 104 | 111 | 3.99 | 1.38 | 1.89 |
| 5.0 % w/v | ± 72 | ±49 | ± 7 | ± 800 | ±14 | ± 10 | ± 7.4 | ± 7 | ± 2 | ± 0.33 | ± 0.25 | ± 0.14 |

*Samples: 1- UHT integral milk supplemented with Fe; 2 - Raw bovine milk; and 3 - UHT integral milk. ^br.s.d. (n = 3).

The use of papain was discharged due to high blank solution. Iron is preferentially linked to organic compounds present in the milk protein phase being liberated by the action of proteolitic enzyme. Calcium and zinc are present mainly in the casein and, depending on the extractor reagent they are solubilized to the aqueous phase compartment. Magnesium is mostly associated with aqueous fraction, probably associated with inorganic species such as citrate, phosphate and carbonate ions.

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