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Time-Domain Nuclear Magnetic Resonance as a Potential Analytical Tool to Assess Thermisation in Ewe's Milk

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Abstract : Some of the artisanal cheeses products of European Countries certificated as PDO (Protected Designation of Origin) are made from raw milk. To recognise potential frauds (e.g. pasteurisation or thermisation of milk aimed at raw milk cheese production), the alkaline phosphatase (ALP) assay is currently applied only for pasteurisation, although it is known to have notable limitations for the validation of ALP enzymatic state in nonbovine milk. It is known that frauds considerably impact on customers and certificating institutions, sometimes resulting in a damage of the product image and potential economic losses for cheesemaking producers. Robust, validated, and univocal analytical methods are therefore needed to allow Food Control and Security Organisms, to recognise a potential fraud. In an attempt to develop a new reliable method to overcome this issue, Time-Domain Nuclear Magnetic Resonance (TD-NMR) spectroscopy has been applied in the described work. Daily fresh milk was analysed raw (680.00 µL in each 10-mm NMR glass tube) at least in triplicate. Thermally treated samples were also produced, by putting each NMR tube of fresh raw milk in water pre-heated at temperatures from 68°C up to 72°C and for up to 3 min, with continuous agitation, and quench-cooled to 25°C in a water and ice solution. Raw and thermally treated samples were analysed in terms of 1H T2 transverse relaxation times with a CPMG sequence (Recycle Delay: 6 s, interpulse spacing: 0.05 ms, 8000 data points) and quasi-continuous distributions of T2 relaxation times were obtained by CONTIN analysis. In line with previous data collected by high field NMR techniques, a decrease in the spin-spin relaxation constant T2 of the predominant 1H population was detected in heat-treated milk as compared to raw milk. The decrease of T2 parameter is consistent with changes in chemical exchange and diffusive phenomena, likely associated to changes in milk protein (i.e. whey proteins and casein) arrangement promoted by heat treatment. Furthermore, experimental data suggest that molecular alterations are strictly dependent on the specific heat treatment conditions (temperature/time). Such molecular variations in milk, which are likely transferred to cheese during cheesemaking, highlight the possibility to extend the TD-NMR technique directly on cheese to develop a method for assessing a fraud related to the use of a milk thermal treatment in PDO raw milk cheese. Results suggest that TDNMR assays might pave a new way to the detailed characterisation of heat treatments of milk.

Keywords : cheese fraud, milk, pasteurisation, TD-NMR
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