Pasteurization and Sterilization of Milk by Supercritical Carbon Dioxide Treatment

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Abstract.

Supercritical (SC) CO_2 treatment of row skim milk was studied in order to improve the shelf-life while maintaining its nutritional and organoleptic properties. To this purpose, experimental laboratory tests were made and an optimal set of operating process parameters were found. In fact, at P = 150 bar, 35 °C ≤ T < 40°C, and CO_2 /Milk feed ratio equal to 0.33, the treatment is significantly more efficient than the traditional thermal pasteurization (HTST technology, at 75÷80 °C for 15÷20 s) made in continuous mode by using a proper thermal exchanger system. Finally, a new process for the SC CO_2 mild pasteurization of whole milk at industrial scale was described and discussed.

Introduction

Thermal pasteurization and sterilization is a well known and old technique for reducing and/or inactivating the microbial charge of food and beverages. However, when the operating temperature is significantly above the ambient value, the product can be damaged as both nutritional (e.g.: degradation of vitamins) and organoleptic (change of taste, aroma or texture of food) properties. Consequently, for more than to decades now, a number of mild/non-thermal technologies were investigated and in some case successfully applied in the food and beverages sector. Among these, high hydrostatic pressure (HHP) and pulsed electrical fields (PEF) are the most diffused ones but both of them suffers of some serious limitation which need still to be solved (Devlieghere et al, 2004; Estrada-Giron et al., 2005). Microfiltration is, at moment, the most commercial diffused mild technology for extending refrigerated milk shelf life. This process, developed by Tetra Pak (Pully, Switzerland) is usually known as Bactocatch process; it and was initially proposed by Holm et al. (1986) and more recently revised by Krabsen et al. (1997) and developed by APV (Aarhus, Denmark), and called Invensys (te Giffel and van der Horst, 2004). Microfiltration of skim milk, often in combination with HTST or ultra-pasteurization (UP) of cream centrifugally removed in advance and/or the retentate, has been used commercially in United Kingdom by Cravendale (a subsidiary of of Arla Foods, Leeds, UK) and in Canada by Natrel (a division of Agropur cooperative, Longueuil, Quebec) who achieved a refrigerated shelf life of 32 d, using a 1.4 µm ceramic membrane filter. Much work has been completed to investigate commercial applications of high pressure carbon dioxide (HPCD) inactivation of microorganisms in food (Garcia-Gonzales et al., 2007) in the last two decades; However, only few research groups have investigated the effects of HPCD treatment on microbial inactivation in milk and, in addition, all such applications involved batch, not continuous or semi-continuous systems

(Werner and Hotchkiss, 2006). The purpose of this was to investigate, at laboratory scale, the quantitative effect of supercritical carbon dioxide ($SCCO_2$) treatment on the shelf life other than on nutritional and organoleptic properties of skim milk, to find an optimal set of operating process parameters, and to develop a continuous $SCCO_2$ process for industrial production of non thermal pasteurized whole liquid milk.

Experimental

The laboratory experimental apparatus used in this work is shown schematically in Figure 1.

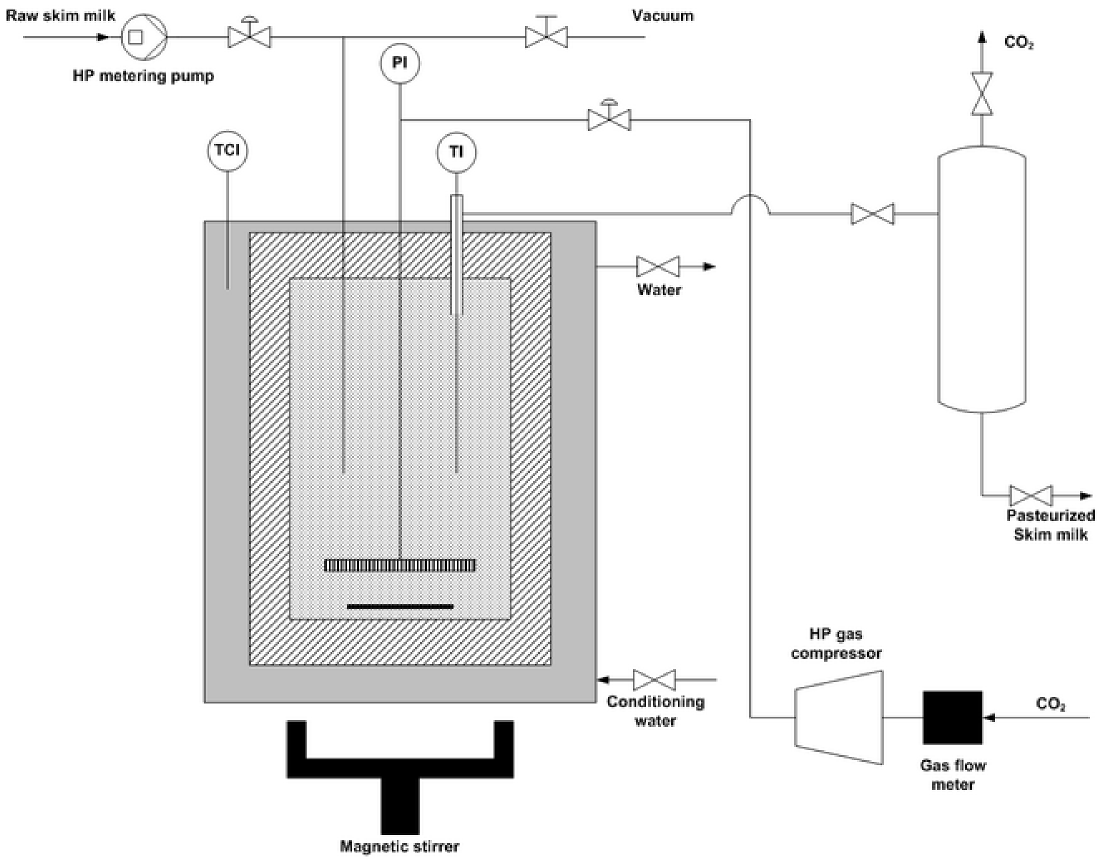

Figure 1. Schematic diagram of the laboratory experimental apparatus

The main component of the system is a 0.2 liters AISI 329 stainless steel autoclave (NOWA-WERKE, Zurich, Switzerland) designed to operate up to 70 MPa and 350 °C; constant operating temperature is attained by a circulating a constant (± 0.1 °C) temperature water stream coming from an automatically controlled thermostat. The three opening on the top of the autoclave are used, as shown, for continuously feeding both the liquid skim milk and the $SCCO_2$ at constant rate obtained by using an high pressure metering pump and an high pressure compressor in combination with a mass gas flow meter (MICRO MOTION D6), respectively. The temperature inside the autoclave was measured by a type J thermocouple which allows one to have continuous temperature readings with an accuracy of ± 0.2 °C. The pressure inside the autoclave is measured by a pressure transducer (Haenni, EDR430) with an accuracy of ± 0.15 % and it is continuously displaced by a digital indicator. A Teflon coated small bar is also inserted into the cell to allow magnetic stirring while, a gas diffuser is attached at the end of the small CO_2 entering pipe. Before starting a single experimental test the autoclave was hermetically closed, sterilized with overheated steam at 12 bar for 0.4 h, thermally conditioned by circulating the water outside, and evacuated. Then, the compressor and the metering pump were switched on to allow the skim milk and the $SCCO_2$ to continuously entering and leaving the reactor. The stream leaving the autoclave is expanded in the indicated vessel in order to separate the gas from the liquid product, which is collected in a sterile glass vessel and putted in a refrigerator. The described system was operated under different residence time, pressure, temperature, and CO_2/skim milk ratio in order to find an optimal set of operating parameters. To this purpose we took advantage of previously reported results on the solubility power of $SCCO_2$ (Di Giacomo et al., 1993; Martinez de la Ossa et al., 1990; Di Giacomo et al., 1991), other than from the value of the density and other physical properties of $SCCO_2$ under different P and T conditions (Gas Encyclopedia, 1976).
Bulk raw whole milk and bulk raw skim milk along with the corresponding HTST pasteurized product, were obtained in the required quantity by the local production plant having a potentiality of 4÷5 m^3 /d. After some preliminary trials it was decided to run each experimental tests for 3.5 h. At the end of each experimental test the processed milk was analyzed by an organoleptic panel made by formally recognized expert, for comparison with a HTST pasteurized reference sample having the same age; the organoleptic test was repeated each to days in a period of 35 days.

Results and Discussion

The selected set of optimal operating parameters is: P=15 MPa; T=35÷38 °C; (CO_2/skim milk feed ratio)=0.30÷0.33; Residence time inside the autoclave=0.25 h. Under this conditions, the shelf life of the $SCCO_2$ pasteurized skim milk is higher than 35 days as resulting from the organoleptic test; in addition, from the same test it came out that the taste of the $SCCO_2$ pasteurized skim milk is significantly better in comparison to the thermally pasteurized reference sample. It is worth to point out that when working at higher value of the pressure the organoleptic properties are damaged since more aroma compound are extracted. It was not possible to evaluate the effect on the nutritional properties of the mild treated sample but it is reasonable to assume that at temperature lower than 38 °C the vitamins and are not damaged.

Process development

Figure 2 show a preliminary industrial process flow diagram developed by using the experimental results reported in the previous section. As can be seen, this process is similar to the Bactocatch process since it includes an homogenization section (OM) and a thermal pasteurization section (AS) for the cream separated centrifugally separated (C) by the whole milk before the $SCCO_2$ pasteurization treatment (CP). In this way it is possible to produce whole mild pasteurized milk characterized by a significantly long shelf life for the refrigerated product. It my be useful to underline that the separation of the cream from the whole milk before the $SCCO_2$ pasteurization treatment is related to the main mechanism of the microorganisms inactivation of the $SCCO_2$ (Garcia-Gonzales et al., 2007).

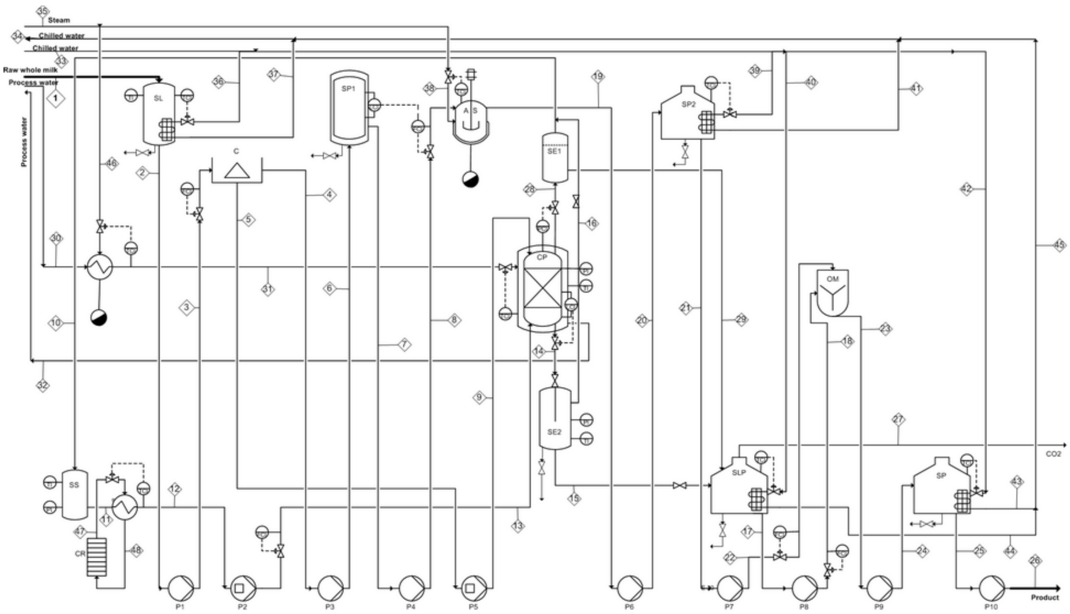

Figure 2. Process flow diagram for industrial production of SCCO2 pasteurized whole milk

Conclusions

Supercritical carbon dioxide treatment of skin milk can be considered as a mild effective pasteurization process since the product obtained in this way is characterized by a comparatively high shelf life, while maintaining the organoleptic properties of the fresh untreated corresponding product. To this purpose, an optimal set of operating parameters must be accurately selected.

This process can also be used in combination with the HTST or with the ultra-pasteurization of cream centrifugally removed in advance to produce mild pasteurized whole milk. In this case, a validation of the whole process at pilot plant scale is recommended along with an accurate feasibility study for the economic implications.

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