DIC Decontamination of Solid and Powder Foodstuffs

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1 Introduction

Stricter safety standards are becoming an increasingly restrictive element in food production. Furthermore, the precautionary procedures adopted in various manufacturing industries to produce healthy foods are very important but in no way sufficient to ensure complete safety from different microbiological contaminations. Indeed, cleaning processes are never perfect and there is an acute need for a specific microbiological decontamination step to be included in the industrial process, even in the most developed countries.

Although many new techniques have been studied, optimized, and sometimes used to protect food from microbiological deterioration, ultrahigh temperature (UHT) is usually considered to be the most valuable decontamination process. Other techniques such as microwaves and ohmic heating sometimes help to improve the performance of this type of operation. Physical treatments, such as pulsed electromagnetic field (PEF), ultraviolet (UV) treatment, and ultrahigh pressure, have been studied, optimized, and applied. Although the use of the broadest possible range of technologies has been successfully implemented with several new types of treatment, their use remains very limited. Chemical additives and ionization are increasingly being rejected by consumers and limited by international standards.

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There are multiple barriers to achieving good decontamination in terms of technical performance (efficiency of microorganism destruction, energy consumption) and an efficient preservation of the attributes and quality of the end product (suitable safeguarding of biochemical, nutritional, and sensory contents). Heat treatment remains the major industrial operation used to destroy microorganisms, and UHT has been a real success in the food industry because it can clearly satisfy these two contradictory constraints. With specific optimization needed for each case, conventional UHT is not limited to pasteurization and also includes sterilization. However, UHT is only used with liquids as materials that can pass through a pump using exchangers for both the heating and/or cooling stages.

A large range of dried foodstuffs such as mushrooms, spices, herbs, onions, garlic, and flour are known to have a high microbial load, sometimes combined with an infestation with insects. This is often the result of traditional methods of harvesting, drying, grinding, storage, etc. Very few relevant decontamination technologies are appropriate for this type of product. Heat destruction of microbiological organisms in solids is faced with several types of difficulty. Conventional exchangers are not appropriate for the kind of heat transfer required. Conventional steam treatments often need long periods of rising and falling temperature. During the heating and cooling stages, a strong temperature gradient is produced and the products may suffer from a lack of homogeneity, which typically involves damage to the end product and reduces its overall quality. Although microwave heating is easy, it is almost impossible to obtain a uniform product and this method is not appropriate for dried food products, whatever their shape and size.

The use of both chemical and room-temperature specific gases such as ethylene oxide or propylene oxide is a long-standing practice. Nevertheless, international standards (European, US, Japanese) are increasingly stringent, mainly with regard to residual chemical molecules generated or introduced into the treated product, which in general are completely banned; ethylene oxide, for example, has been prohibited in France since 1990. Consumer attitudes are significantly reinforcing this tendency.

Nuclear gamma irradiation has been considered for a very long time to be the best and most convenient decontamination method, mainly for solid and/or powder foodstuffs. This treatment has proved to be highly effective and very convenient since the product can be treated in its airtight packaging, avoiding any recontamination. The dose for food depends on the desired effect: “low” dose irradiation (50–150 Gy) does not decontaminate and is used only to inhibit sprouting of potatoes and onions. Food sterilization (e.g., precooked meals) requires much higher doses (10–50 kGy). Nevertheless different authors have noted some negative effects of such treatment. Because of the radiation energy, ionization can remove electrons from atoms and break molecular bonds, leading to the formation of highly reactive free radicals. New molecules could thus appear in the food as a result of chemical recombination. The irradiation of lipids causes the formation of cyclobutanones whose toxicity is well known. Such chemical toxicity has been shown to have very disturbing effects (including chromosomal damage).

This type of irradiation treatment eliminates microorganisms in food, including those with useful features. However, some acids and vitamins (including A, B1, B6, B12, C, E, K, PP, and folic acid) can be damaged by radiation treatment, depending on the dose and the radiosensitivity of the molecules. High-dose irradiation destroys microorganisms but does not remove the toxins previously produced, which are often responsible for many foodborne illnesses. Moreover, some authors have highlighted the risk of a particular mutation which can be induced by irradiation in insects and bacteria. In addition to these scientific and technical aspects and despite a substantial marketing effort deployed by the industry to attest to the safety of irradiation (proved only for doses up to 10 kGy), ionizing radiation suffers from a very bad image due to the confusion between radiation and radioactivity. Rejection by consumers has been particularly reinforced since the introduction in 2001 of mandatory labeling of all products processed by ionizing radiation. These elements and the relatively high cost of the equipment explain its very low level of approval (Allaf et al. 2011).

2 DIC as an UHT Decontamination

"Instant controlled pressure drop" (DIC) is typically presented as a specific UHT treatment for solid and powder foods. Generally, heat treatment by DIC is a short controlled process carried out at high temperature (Fig. 1).

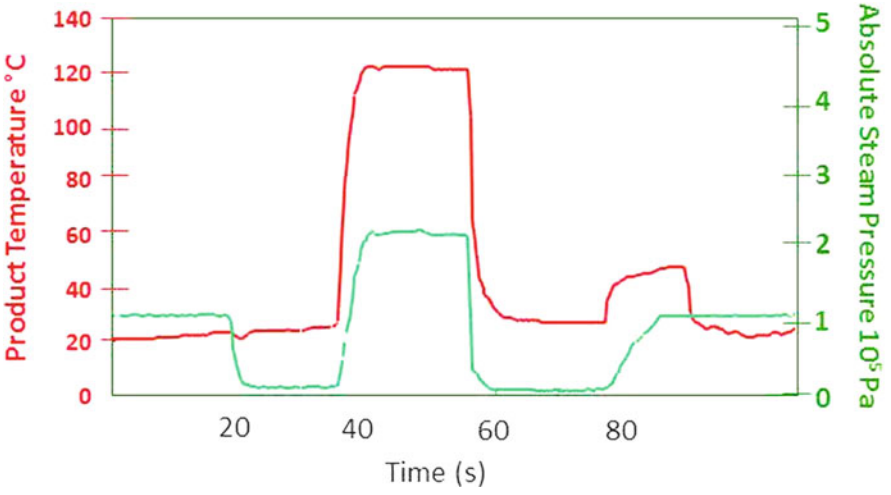

Fig. 1 A DIC treatment cycle: change in pressure and temperature versus time and various steps

The initial heating of the product can be substantially intensified and the time during which the temperature is rising can be radically reduced, since an initial vacuum step is inserted just before injecting high temperature–high pressure steam. The close contact between the steam and the surface of the product is indispensable to bring the product temperature to the same level as the steam temperature within a few seconds. The need to heat very rapidly can then "easily" be achieved. Cooling is obtained through the use of an instant controlled pressure drop towards a vacuum and can be measured on a 20/200 ms time frame. With this type of cooling it is possible to lower the temperature by decreasing the vacuum pressure. Typically, for DIC the vacuum is at about 5 kPa and the equilibrium temperature about 35 °C. Recent work has demonstrated the possibility of significantly reducing the temperature of the product to below the equilibrium level. This treatment has been studied in depth and the authors showed that it had a similar thermal effect to UHT for liquids. The treatment temperature is usually between 100 and 150 °C, while treatment time can be limited to 5 s. Furthermore, two other thermal stress impacts (short-time heating and instant cooling) seem to be very effective in the destruction of microorganisms. The instant autovaporization also induces micromechanical constraints that act on the microorganism cell walls and more specifically on the spore wall.

Indeed, specific research on the impact of instant pressure drop on cell structures and other biological structures is both of scientific interest and has an obvious technological importance. Products are initially placed under a vacuum and then saturated or superheated steam is injected at a fixed pressure which rises to an absolute pressure of 7 or 8 bar. This results in very rapid heating, mainly through condensation on the inner (fractal) surface of the product. With 5 mm thick dry products this can be achieved in less than 3 s. The heating time is completely controlled by inserting an intermediate vacuum stage at around 5 kPa of absolute pressure.

After the heating stage, which is usually carried out between 100 and 150 °C for 5–60 s, a second vacuum stage is immediately performed and lasts less than 100 ms, i.e., a decompression rate higher than 5 MPa/s. Thanks to autovaporization, the temperature of the product drops abruptly to very low levels, which must be lower than the equilibrium temperature of the water, which in our case is 33 °C.

Other versions of this process consist in achieving proper heating using high temperature adiabatic airflow (or inert gas). Moreover, heating can be achieved through contact with hot plates or even by microwaves. Coupled with vibration or mixing, the products attain the treatment temperature in a more uniform way. The application of an instant pressure drop towards a vacuum generates instant cooling by autovaporization, thus partly removing the water in the product.

This treatment perfectly reflects a UHT-type heat treatment but is here applied to solids instead of liquids. Thus, studies conducted by Debs-Louka et al. (1999) on the DIC-mediated microbial destruction of *Bacillus stearothermophilus* spores gave a value of $D_{121,1\ ^\circ C}$=2.6 min (Fig. 2) and a thermoresistance z of 7.6 °C, instead of 4.2 min and 8.8 °C, respectively (Le Jean et al. 1994).

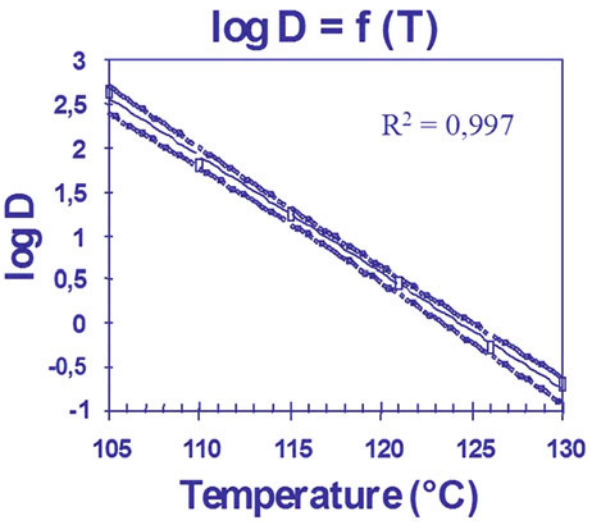

Fig. 2 Change in log *D* versus temperature for *Bacillus stearothermophilus* spores

3 DIC Thermomechanical Impacts

It was also possible to prove that the effect of the instant pressure drop in DIC was not confined to the impact of abrupt cooling. A thermomechanical effect that may lead to the explosion of microorganism cells (spores or vegetative forms) also occurs (Fig. 3).

Indeed, the higher the amount of "steam" generated by autovaporization within the cell and the shorter the pressure drop time, the more efficient the mechanical effect is.

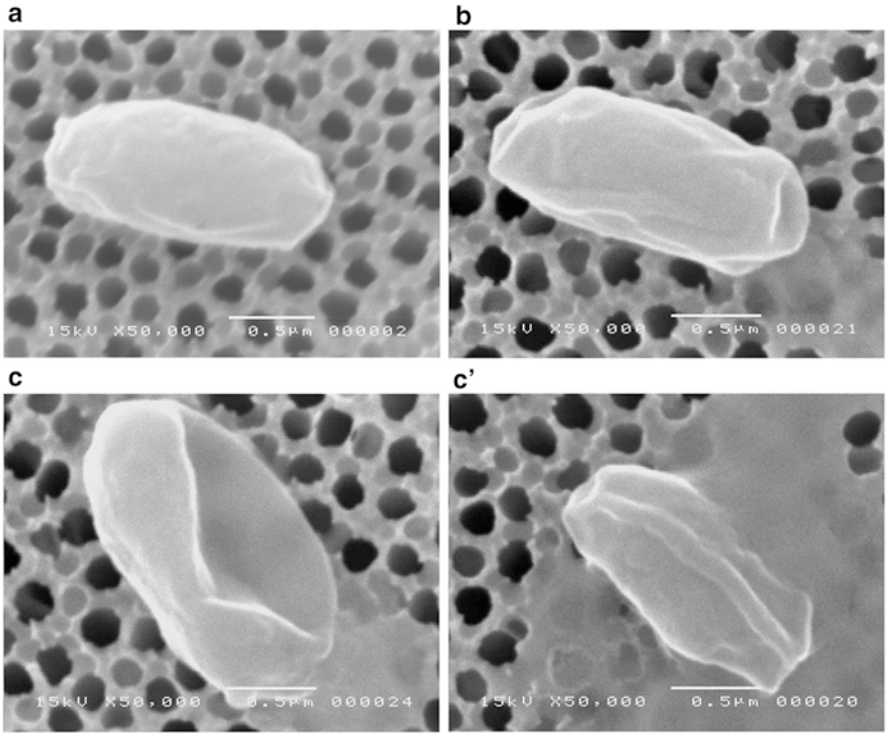

Fig. 3 Structure of *Bacillus stearothermophilus* spores: (*A*) Natural; (*B*) Heat treated at 130 °C for 30 s without a pressure drop; (*C and C'*) DIC treated at 130 °C for 30 s with an instantaneous pressure drop

4 Gas Mechanical Effect

Besides water, other molecules can be considered. The choice of molecule is closely related to its potential mechanical action, which is correlated to the difference in magnitude between the internal and the external pressure generated when the pressure instantaneously drops towards a vacuum; this normally depends on the rate of the pressure drop.

The choice of carbon dioxide was dictated by the relatively high dissolution capacity of this gas in microorganism cells during the high-pressure stage. For each fluid used the most important point was its capacity to generate a force capable of cracking and even breaking the cell walls. The treatment conditions were defined as being outside the supercritical range in order to avoid extraction consequences (Fig. 4). Other experimental approaches were carried out to identify the effect of the product's moisture content on the destruction of microorganisms by instantaneously dropping the pressure of CO_2.

Thus, experiments were conducted with *E. coli* and *S. cerevisiae* to investigate the influence of moisture content. Lin (1984) found that moisture content was vital to the antimicrobial action of CO_2. However, treatment carried out with carbon dioxide at 4 MPa for 195 min followed by an instant controlled pressure drop, with a moisture content of between 37 and 75 % dry basis, gave the same rate of destruction for the two microbial strains. Nevertheless, moisture content of about 6 % db was too low for inactivation.

Moisture content (% db dry basis)	CO_2 conditions (pressure–time)	log (N/N_0) (*Escherichia coli*)	log (N/N_0) (*Saccharomyces cerevisiae*)
37 % db—75 % db	5 MPa—300 min	−4.8	−4.3
6 % db	5 MPa—300 min	−0.09	−0.57

In the near future, further studies on the impact of high temperature will be carried out to investigate different aspects of high pressure/instant pressure drop with carbon dioxide.

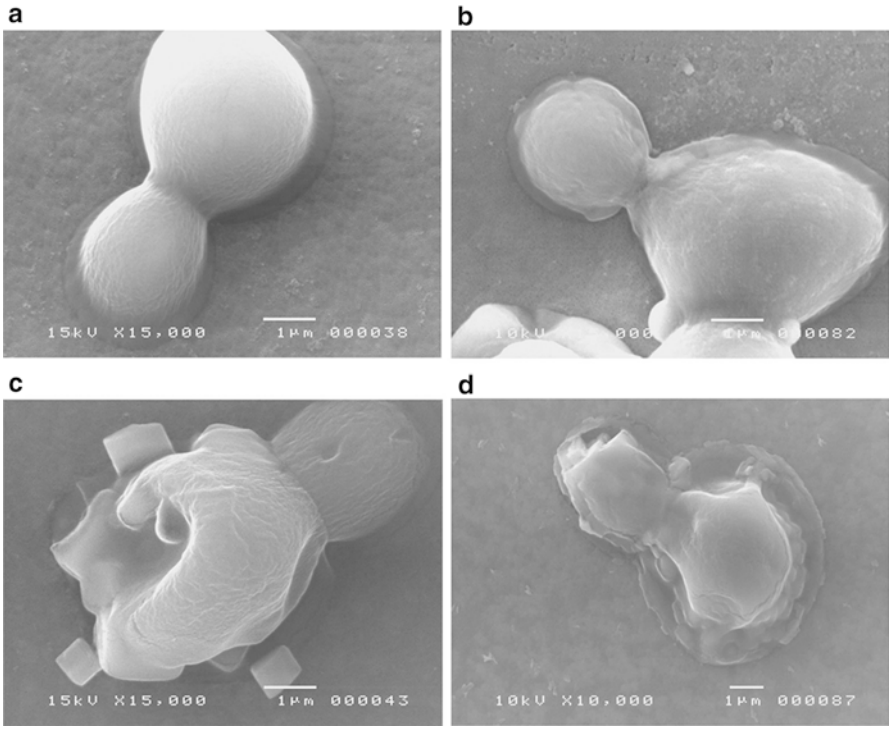

Fig. 4 *Saccharomyces cerevisiae* cell structure: (*A*) Natural; (*B*) Heat treated at 130 °C for 30 s without a pressure drop; (*C*) DIC treated at 105 °C for 30 s with an instantaneous pressure drop towards a vacuum (5 kPa; $\Delta P/\Delta t > 5$ MPa/s); (*B'*) High-pressure carbon dioxide treatment at 55 bar for 5 h, without a pressure drop; (*C'*) High-pressure carbon dioxide treatment at 55 bar for 5 h, and ambient temperature, with a pressure drop towards a vacuum (5 kPa; $\Delta P/\Delta t > 5$ MPa/s)

5 DIC Decontamination: Industrial Applications

Industrial applications have mainly consisted in adopting thermomechanical steam destruction of microorganisms while maintaining different parameters of food quality (texture, flavor, vitamins, protein activities, etc.). Research carried out by Allaf et al. (1998) and Debs-Louka et al. (1999) identified the different effects of this treatment and the well-established "temperature–time" couple for the level of decontamination. By quantifying the effect on various quality parameters, it was possible to obtain a very relevant and multidimensional optimization.

The major part of the industrial work using DIC treatment was subsequently carried out by the ABCAR-DIC process company. A wide-ranging variety of solids and powders, such as mushrooms, fruits, vegetables, meat and seafood, algae and microalgae, spices, and ginger, were effectively treated. The effect of decontamination by DIC has therefore been optimized at the industrial scale, according to various constraints related to the product and its requirements in terms of quality, the microorganisms that must be eliminated, and those that should be preserved.

6 Multi-cycle DIC Decontamination

The impact of the number of DIC cycles (various pressure drops towards a vacuum for the same total processing time) was quantified for dairy powder decontamination (Fig. 5; Table 1). The specificity of DIC treatment is related to the ability to define the degree of decontamination across the triumvirate "temperature/treatment time/number of pressure drops" instead of the conventional torque "temperature–treatment time." The impact of this specificity is immediate in terms of the quality of the finished product.

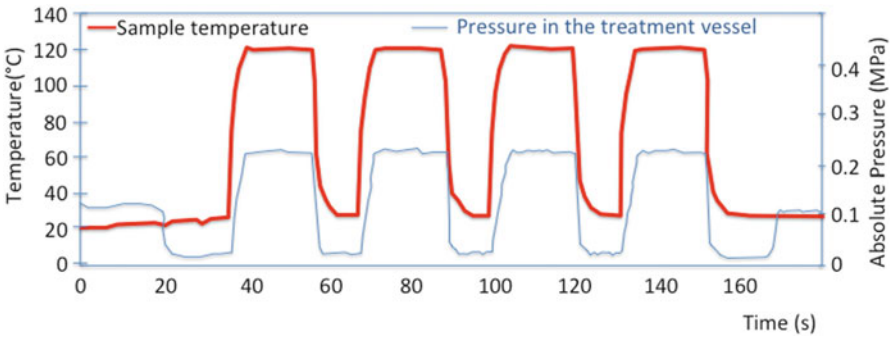

Fig. 5 Multi-cycle DIC decontamination treatment

Table 1 Multi-cycle DIC decontamination treatment conditions using hot air at 0.3 MPa, a vacuum of 5 kPa, and a pressure drop of about 1.5 MPa/s

Hot air	Temperature (°C)	Time (s)	Number of cycles	Time per cycle (s)
B1	135	360	6	60
B2	150	300	6	50
B3	135	420	6	70
B4	135	300	6	50
B5	144	360	8	45
B6	135	300	10	30
B7	135	300	6	50
B8	144	240	8	30
B9	144	300	4	75
B10	135	300	6	50
B11	144	240	4	60
B12	126	360	8	45

Cereus spores were inoculated at 10^4 CFU/g and analyses were performed in terms of both spores and vegetative forms, before and after DIC treatment. Statistical analysis (Statgraphics Plus) was mainly carried out on the decontamination ratio, which is the percentage of germs eliminated:

$$\text{Decontamination ratio (DR)} : \text{DR} = \frac{N_o - N}{N_o} \quad (1)$$

As the experiments were undertaken with an instant release of pressure, from high temperature towards the equilibrium temperature of water/vapor at 5 kPa, the operation was carried out in the "explosion conditions" defined by Lin et al. (1991, 1992). In the cases we studied, only 20–200 ms were necessary to reach the complete vacuum stage from 0.3 MPa ($\Delta P/\Delta t$ from 1.5 to 15 MPa/s) within the processing vessel. When the same treatment conditions were used but with a decompression time of 12 s ($\Delta P/\Delta t = 25$ kPa/s), no explosion effect occurred. Statistical analysis carried out on the results concerning the inhibition of vegetative and spore forms indicated that with multi-cycle DIC, the effects of processing temperature and total thermal processing time (t) were the most relevant parameters. However, the number of cycles (C) had a non-negligible effect. It is worth noting that the higher the T, t, and C, the greater the direct impact of multi-cycle DIC decontamination (Figs. 6 and 7).

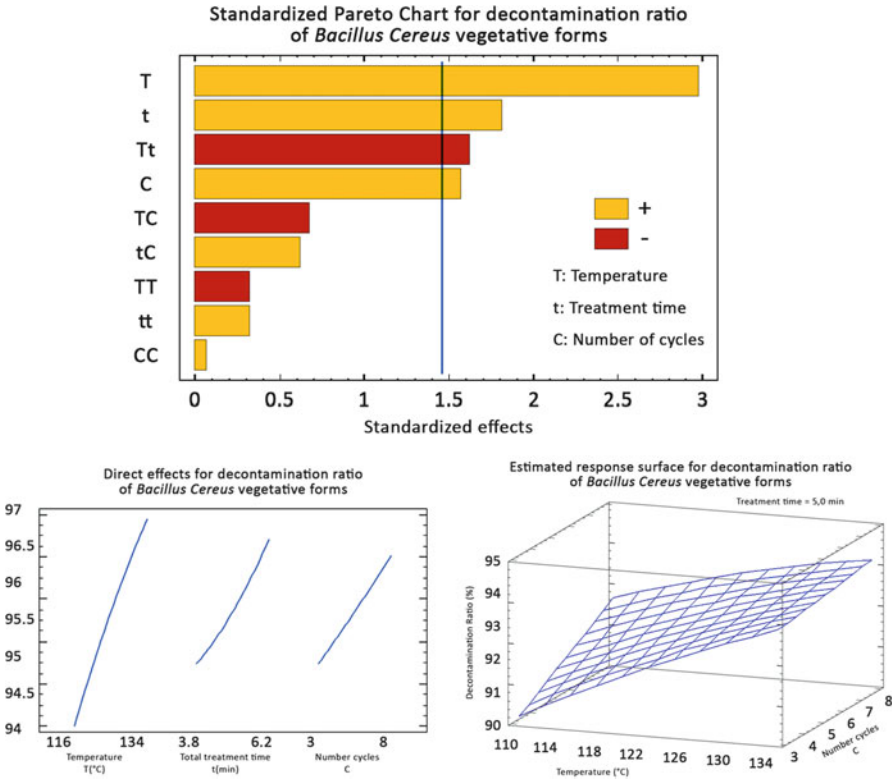

Fig. 6 Pareto chart, trends of main effects, and response surfaces of pressing temperature T (°C), total thermal treatment time t (s), and number of cycles (C) as multi-cycle DIC operating parameters from a five-level central composite rotatable RSM experimental design with ASR vegetative forms

It was then possible to establish empirical models of the MC-DIC decontamination ratio of both ASR vegetative and spore forms versus the DIC processing parameters, with $R^2 = 60.2$ % and 76.77 %, respectively:

$$\mathrm{DR}_{\text{vegetative-ASR}} = -806 + 8T + 73t + 14C - 0.012T^2 - 0,64Tt - 0.13TC + 0.67t^2 + 0.91tC + 0.03C^2$$

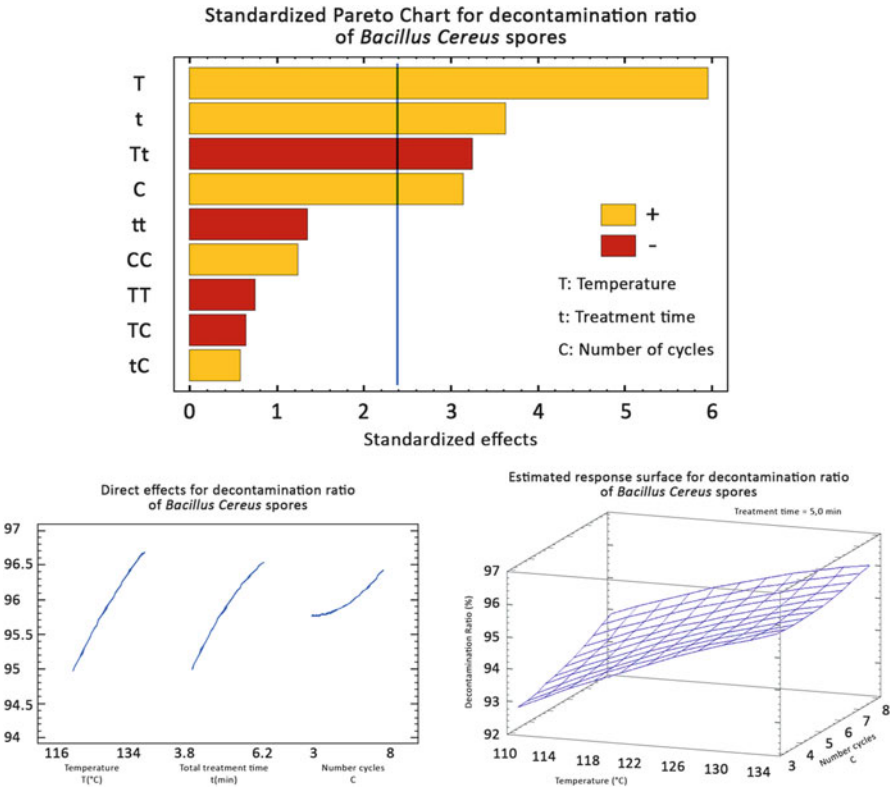

Fig. 7 Pareto chart, trends of main effects, and response surfaces of pressing temperature T (°C), total thermal treatment time t (s), and number of cycles (C) as multi-cycle DIC operating parameters from a five-level central composite rotatable RSM experimental design with ASR spores

$$\mathrm{DR}_{\text{spores-ASR}} = 1,365 + 13T + 163t + 6C - 0.02T^2 - 1,1Tt - 0.1TC - 1.97t^2 + 0.66tC + 0.45C^2$$

These two models were used to optimize the multi-cycle DIC treatment parameters in order to reach the highest decontamination ratio while taking into account the preservation of quality. The higher the operating parameters T, t, and C, the higher the decontamination ratio. However, it is worth noting that the effect of C is specific because its positive impact on decontamination does not imply any thermal degradation of quality.

7 Conclusion

Instant controlled pressure drop (DIC) technology can be defined as a highly appropriate UHT-type decontamination process that can be applied to powders and other dry solid biological materials. This operation also involves thermal stress by both heating and, to a much greater extent, instant cooling. Another stress is induced by vapor constraints acting on the cell walls due to the rapid drop in pressure. This usually induces an explosion of the cell walls of both the vegetative and the spore forms.

Because of these different thermal and mechanical stresses, industrial uses of DIC are very effective for a wide variety of very sensitive biological products. Decontamination is usually optimized according to the product quality to be obtained. Since the number of cycles is a relevant operating parameter, competing with temperature and thermal processing time, multi-cycle DIC treatment is an increasingly appropriate and convenient industrial decontamination process. DIC reactors are currently operating at laboratory, pilot, and industrial scales. Thus, there are several infrastructure models with different features and capabilities; the energy consumption has been calculated to be 0.110 kWh per kg and per cycle.

This work described the identification and industrial uses of a thermomechanical destruction of microorganisms, which is mainly valid for solid products or powders. DIC can advantageously replace conventional processes in this field where many new treatments, such as radiation (γ, ultraviolet, acoustic) or mechanical (UHP, ultrasound) treatments, when possible, have only had limited use.

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