PAPER

HIGH PRESSURE STABILIZATION OF ORANGE JUICE: EVALUATION OF THE EFFECTS OF PROCESS CONDITIONS

STABILIZZAZIONE DEL SUCCO D'ARANCIA A MEZZO DI ALTA PRESSIONE: VALUTAZIONE DELL'EFFETTO DELLE CONDIZIONI DI PROCESSO

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ABSTRACT

A laboratory scale apparatus was designed, set up and tested to investigate the effects of high hydrostatic pressure on the stabilization of orange juice, whose quality is dramatically reduced by the classical thermal preservation processes used in the food industry. An evaluation of the microbial activity as well as of the chemical composition of orange juices processed at different pressure levels and operating times was conducted and compared with that of fresh juice. The overall quality of the orange juice processed at high pressure was excellent and the refrigerated

RIASSUNTO

È stato progettato, realizzato e messo in esercizio un impianto da laboratorio innovativo per la stabilizzazione di alimenti ad alta pressione. La sperimentazione è stata condotta su campioni di succo di arancia, prodotto la cui qualità è fortemente danneggiata dai trattamenti termici tradizionalmente utilizzati nell'industria alimentare per la sterilizzazione di succhi di frutta. Sono state condotte determinazioni sull'attività microbiologica, sulla composizione chimica nonchè sulle caratteristiche reologiche e sul colore dei prodotti trattati. I risultati dimostrano che i tratta-

- Key words: high pressure pasteurization, orange juice. -

shelf life was not less than two months, provided that a minimum pressure level of 3500 bar (process time 1 min and temperature 30°C) was attained.

menti ad alta pressione mantengono pressocché inalterate le caratteristiche del prodotto fresco e che la conservazione del prodotto in condizioni refrigerate può essere protratta fino a due mesi. Il livello minimo di pressione che consente di ottenere la stabilizzazione del succo di arancia è pari a 3500 bar (tempo di trattamento 1 min, temperatura 30°C).

INTRODUCTION

The utilization of very high pressure for the production of special materials with particular characteristics is very well known in the metallurgical as well as in the chemical and ceramics industry. This technology has been recently proposed to produce sterilized foodstuffs with a long shelf life (HAYASHI, 1991; KNORR, 1992).

These products are presently obtained by means of thermal processes at high temperatures, which eliminate the possibility of microbial spoilage and reduce the enzymatic activity. Even though thermal treatments ensure the so-called commercial sterility of foods and extend their availability on the market by several months, they affect the quality of the products.

In fact, due to the loss of thermolabiles and thermosensitive components responsible for the sensory and nutritional properties of foods, the quality of sterilized foods differs very much from that of fresh ones. In particular, the aroma, vitamins and volatile components of sterilized products are dramatically influenced by thermal treatments.

Because the attainment of high and consistent quality is one of the challenges of the food industry, mild preservation techniques able to retain the initial quality of foods are being considered with interest by food processors and the use of high cost technologies can thus be proposed.

Among these, high pressure sterilization has been utilized in the last decade and recently also on the industrial scale (TRAFF and BERGMAN, 1992). The process is carried out in a purely mechanical device, operated as a press, which applies pressure to the food through an intermediate liquid medium. The sample is packaged in a flexible container. In principle the system is very simple, but the process is very expensive due to the high cost of the pressure vessel.

The vessel diameter increases with increase in the available internal volume, the pressure level and the number of operation cycles, which all add to the total cost. The high cost is also related to the intrinsic nature of the operation, which can be carried out only batchwise.

For these reasons, this technique can only be considered for those products with high added value whose selling prices can include the cost of the operation with full acceptance by the consumers. An increase in taste, flavour and nutritional value of products would be appreciated by consumers who would then be willing to pay a premium price for the increased quality level of the food.

Many studies have been conducted on the effects of pressure on different

foodstuffs (HAYASHI et al., 1989; HORIE et al., 1990, SHIGEHISA et al., 1991; ROVERE et al., 1993). On the basis of the results obtained so far it can be concluded that the effectiveness of pasteurization by means of high pressure is strictly related to the nature of the foodstuff, i.e. pH, chemical composition, physical structure, and kind of contaminants in the fresh product. In fact, experimental results on spores demonstrate that hydrostatic pressure levels as high as 10,000 bar are ineffective in reducing and killing this kind of bacteria (TAKI et al., 1990).

This paper reports the results obtained using a laboratory scale high pressure apparatus to stabilize orange juice. Orange juice was chosen because it was considered that a non-thermal sterilization process could significantly improve its quality (OGAWA et al., 1989; TAKAHASHI et al., 1993).

After high pressure treatments microbiological activity and chemical composition were determined to demonstrate the effectiveness of this process and to study the effect of pressure on colour, texture, aroma and nutrients as well as on the shelf life of the processed product.

MATERIALS AND METHODS

Apparatus

The experimental autoclave was designed to achieve an increase in pressure of the intermediate liquid outside the pressure vessel by means of two pumps in series. This is a modification of the conceptual design of the commercially available apparatus. In fact, in commercial units the pressurization of the liquid medium is obtained by direct or indirect compression (MERTENS and DEPLACE, 1992).

a) Direct compression: the lid of the high pressure vessel, driven by a low pressure pump, moves like a piston and transmits the pressure directly to the liquid medium.
b) Indirect compression: a high pressure intensifier pumps the liquid medium into the closed high pressure vessel until the desired pressure level is reached.

Both direct and indirect methods have an intrinsic limit: they do not allow the application of fast pressure changes.

The proposed apparatus consists of an autoclave with an internal volume of 250 mL, which can be pressurized up to 7000 bar. The pressure vessel is connected with a high pressure tube to the two diaphram air operated pumps.

The intermediate liquid is distilled water and flows from the reservoir to the autoclave through the two pumps, which pressurize the liquid up to the pressure level of interest.

On the other side of the pressure vessel a high pressure tube closed by means of an expansion valve allows pressure to be released to the atmosphere at the end of each run. The valve can also be operated to obtain pressure fluctuations inside the vessel during operation.

Fast changes of the pressure level can be applied in the unit. This is to determine the effect of the simultaneous application of hydrostatic pressure and dynamic pressure fluctuations on the stabilization of food samples.

A diagram of the experimental apparatus is shown in Fig. 1. Before pressurization, the autoclave is put in a safety box. A pressure gauge gives the reading of the pressure in the circuit through a strain gauge transducer and the instantaneous value of pressure is also measured and recorded on a chart recorder. Pressurization cycles can be programmed by means of a process computer which also controls all safety devices, such as the automatic locking of the safety box and the alarms. Also the temperature of the intermediate liquid can be measured and recorded during experiments.

Orange juice samples

The samples to be processed were obtained by thawing frozen red orange juice. The thawed juice was filtered to remove suspended particles, packed in plastic pouches and thermally sealed. Some pouches were introduced into the autoclave and processed at the pressure level desired and the remaining samples were used as controls.

After processing, samples were stored in cold conditions at a temperature of 8°C to determine the shelf life and were analyzed every two weeks.

The process conditions investigated were as follows:
pressure level: 2000, 3000, 3500, 4000, 5000 bar
process time: 1 min, 15 min
process temperature: 30°C.

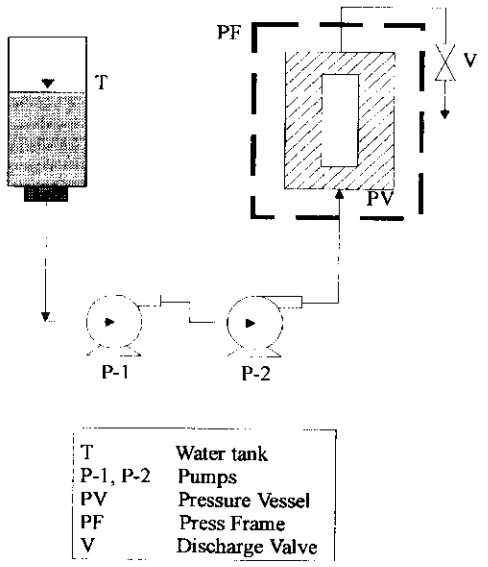

Fig. 1 - Diagram of the experimental apparatus.

Chemical and microbiological determinations

Fresh and processed samples were analyzed with standard methods to determine microbiological activity, chemical composition, pH, colour, aroma and viscosity.

Methods for specific chemical determinations were the following:

A Finnigan Magnum Gas Chromatograph-Mass Spectrometer (Finnigan, San Fernando, CA, USA) fitted with a DB-5 column was used to determine the aroma components of the untreated and processed juice samples. The internal standard was cyclohexanol. Samples were prepared according to a technique proposed in the literature for mandarin juice (TAKAYASHI et al., 1993).

Ascorbic acid was determined using an HPLC, HP 1050 (Hewlett-Packard Co., Avondale, PA, USA), fitted with a Vydac column by Pharmaceutical and a Gilson 115 UV detector, tuned to a wavelength of 245 nm. The mobile phase was a 2% aqueous solution of ammonium dihydrogen phosphate, adjusted to pH 2.8 by addition of phosphoric acid.

The same apparatus, fitted with a C18 column with a linear gradient of acetonitrile/methanol/methylene (80:15:5) and the UV detector tuned to a wavelength of 450 nm, was used for the determination of carotenoids.

Methodology for both ascorbic acid and carotenoids, including sample preparation, was set up according to the suggestions of a recent paper on analytical characteristics of orange juice (MARINI and BALESTRIERI, 1994).

Organic acids were determined according to the methodology described in a recent paper on the measurement of organic acids in fruits (SACCANI et al., 1995).

Carbohydrates were determined according to the official Italian methods for vegetables preserves (G.U., 1989).

Colour parameters were determined by means of a Minolta CR200 reflectance spectrophotometer (Minolta Co., Osaka, Japan), directly focused to a transparent flat wall vessel containing the sample. This device allowed the determination

Table 1 - Results of microbiological analysis of fresh orange juice and juices treated at various pressures (processing time 1 min). n=not detectable.

	Yeasts c.f.u./g	Moulds c.f.u./g	*Lactobacilli* c.f.u./g	*Streptococci* c.f.u./g
Fresh juice	10^2	10^2	2*10	10
2000 bar	< 10	< 10	< 10	n
3000 bar	< 10	< 10	< 10	n
3500 bar	n	n	n	n
4000 bar	n	n	n	n
5000 bar	n	n	n	n

of the brightness parameter L as well as the a* and b* parameters, which indicate colour and colour saturation, respectively. Rheological properties were measured with an automatic rotational viscosimeter with coaxial cylinders (Couette type) Rheometrics RFS2 (Rheometrics Inc., Piscataway, N.J., USA).

Microbiological analyses were performed on samples of untreated and processed juice previously homogenized. Each analysis was repeated at least three times.

Test conditions were the following:

Moulds and yeasts: yeast medium agar DIFCO (DIFCO, Detroit, MI, USA), counts after 120 h of incubation at 28°C;

Lactobacilli: MRS OXOID (Unipath Ltd, Basingstoke, U.K.), counts after 48h of incubation at 42°C in aerobic as well as in anaerobic conditions;

Streptococci: M17 OXOID (Unipath Ltd, Basingstoke, U.K.), counts after 48h of incubation at 42°C.

RESULTS

Results of microbiological analyses of samples processed at different pressure levels indicate that the inactivation of contaminating microorganisms was attained at a minimum pressure level of 3500 bar (Table 1).

Storage in cold conditions (8°C) allowed the orange juice to be preserved for at least two months.

No significant effect due to the processing time was noted. In fact, the inactivation of non-spore forming bacteria, yeasts and moulds was achieved even at a processing time of one minute. The processing time was evaluated since the desired pressure level was reached and thus did not include the time for pressure rise and relief.

Results of chemical analyses are reported in Tables 2, 3 and 4. It clearly appears that no substantial modification in the composition of vitamins, sugars and organic acids occurred at any of the pressure levels tested.

Aroma components were determined by means of gas-mass chromatography. Table 5 only reports the volume concentrations of δ-limonene, which is considered to be the least stable aroma component and is subject to a dramatic reduction in thermal sterilization (TAKAHASHI et al., 1993). δ-limonene content of processed juice essentially remained the same as in the fresh samples. Other aroma components, such as γ-terpinene, p-cymene, linalool, decanale and myrcene were determined, but no significant modifications due to pressure were detected. Also the pH of the orange juice was measured. As shown in Table 5, no appreciable modifications occurred.

The effect of pressure on the colour of orange juices was also evaluated by means of chemical analysis and spectrophotometry. Table 5 shows the con-

Table 2 - Effects of pressure treatments (processing time 1 min) on various vitamins.

	C mg/L	B6 ppb	B2 ppb	Niacine ppb	B1 ppb
Fresh juice	545	1341	295	5674	1983
2000 bar	553	765	290	6024	1823
3000 bar	551	1227	238	5757	1781
3500 bar	537	1149	220	5541	1577
4000 bar	558	1114	290	5747	1573
5000 bar	556	1376	283	5137	1753

Table 3 - Effects of pressure treatments (processing time 1 min) on organic acids.

	Malic acid g/L	Citric acid g/L	Isocitric acid g/L
Fresh juice	0.99	13.33	180
2000 bar	0.93	12.85	156
3000 bar	0.93	12.74	170
3500 bar	0.91	12.65	177
4000 bar	0.97	13.03	182
5000 bar	0.95	12.85	169

Table 4 - Effects of pressure treatments (processing time 1 min) on sugars.

	Sucrose g%	Fructose g%	Glucose g%
Fresh juice	4.65	2.58	2.06
2000 bar	4.64	2.57	2.11
3000 bar	4.80	2.70	2.19
3500 bar	4.69	2.09	2.61
4000 bar	4.73	2.16	2.67
5000 bar	4.72	2.64	2.13

Table 5 - Effects of pressure treatments (processing time 1 min) on pH, aroma and colour indexes.

	pH	δ-limonene %v/v	β-carotene mg/100 mL	Total carotenoids mg/100 mL
Fresh juice	4.27	0.030	0.27	5.1
2000 bar	4.28	0.030	0.25	5.0
3000 bar	4.28	0.028	0.25	5.0
3500 bar	4.29	0.028	0.25	5.0
4000 bar	4.29	0.028	0.24	5.0
5000 bar	4.29	0.028	0.24	5.0

centration of β-carotene and total carotenoids in samples processed at different pressures. It can be concluded that pressure did not affect significantly the concentration of the colour components. This is also confirmed by the direct colour measurements through spectrophotometry. Fig. 2 shows values of the brightness parameter L, the hue angle parameter a* and the colour saturation parameter b* of fresh and processed juice as a function of pressure. The same parameters were also determined on processed samples as a function of storage time, as shown in Fig. 3 at the pressure of 3500 bar. The brightness L of treated juices did not change significantly with pressure or with storage time. The same trend was also exhibited by a* and b*.

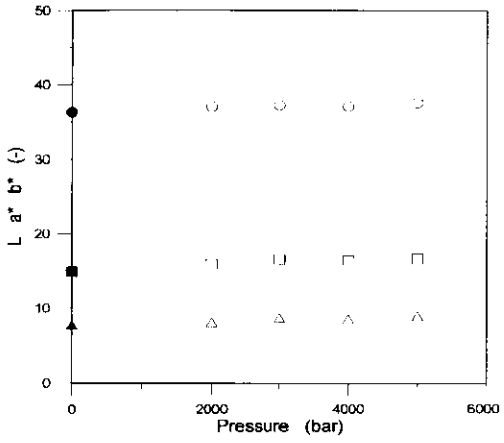

Fig. 2 - Brightness L (O), hue parameter a* (❑) and colour saturation b* (Δ) of fresh and processed juices (processing time 1 min, temperature 30°C) as a function of pressure. Solid symbols: fresh juice. Open symbols: processed juices.

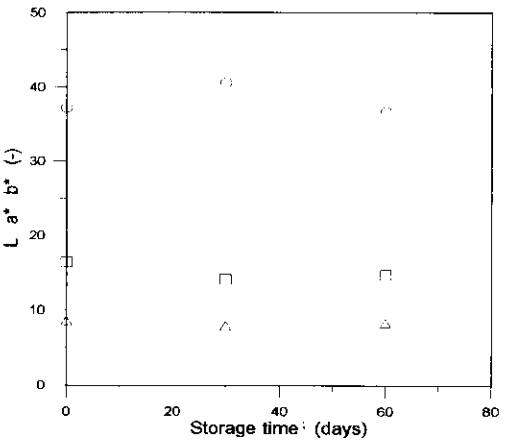

Fig. 3 - Brightness L (O), hue parameter a* (❑) and colour saturation b* (Δ) of processed juices (pressure 3500 bar, process time 1 min, temperature 30°C) as a function of storage time.

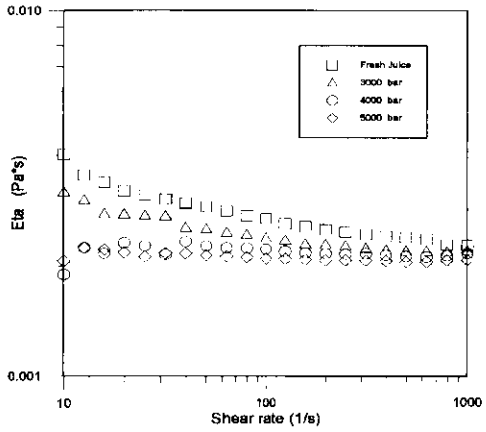

Fig. 4 - Non-Newtonian viscosity of fresh and processed juice (processing time 1 min) as a function of shear rate.

The effect of the high pressure process on the rheological behaviour of the juices was also determined. Results, reported in terms of the so-called non-Newtonian viscosity i.e. the ratio between shear stress and the corresponding velocity gradient, are shown in Fig. 4. Fresh juice showed a pseudoplastic behaviour. Pressurization caused a reduction in the non-Newtonian viscosity, giving rise, at the highest pressure levels, to a constant value of the latter parameter according to the behaviour of Newtonian fluids.

CONCLUSIONS

The successful operation of the experimental apparatus designed by the authors demonstrates the applicability of a conceptually new pressurization technique, based on the use of on-line pumps in the circuit of the intermediate fluid. The main features of this technique are the achievement of fast pressure rise and relief and the high reliability, due to the utilization of simple mechanical components, such as pumps and valves. This apparatus compares favourably with commercial pressurization devices of the present generation based on mechanical pressure intensifiers coupled to the autoclave. They are intrinsically slow in operation, must be recharged after each stroke and require specific mechanical components, tailored to the apparatus.

In the study of the effect of high pressure on the characteristics of foodstuff,

the proposed apparatus seems very promising in order to test the effect of fast pressure variations as well as that of static pressure. In its present version, the apparatus is also able to produce medium frequency pressure fluctuations of the amplitude of 200 bar, superimposed to any pressure level below 7000 bar.

Experiments performed thus far, carried out mainly under static conditions, demonstrated that the high pressure treatment is effective in achieving the preservation of orange juice. This product was fully stabilized for commercial purposes at a pressure not lower than 3500 bar. At this pressure a processing time of one minute was sufficient to inactivate microrganisms for commercial stabilization. The effect of process time, in the pressure range investigated, was almost undetectable.

The stabilized juice had a shelf life of at least two months in refrigerated conditions (8°C), as proved by periodical microbiological analysis. The quality level of the untreated product was fully maintained, with respect to the main chemical components, vitamins, sugars and organic acids.

Further work is needed to clarify the combined effect of process time and pressure as well as to investigate the effect of pressure fluctuations.

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REFERENCES

G.U. 1989. Italian Official Gazette n. 168 20/7/1989. Metodi Ufficiali per le Conserve Vegetali.

Hayashi, R. 1991. Progress of high pressure use in food industry. Proc. of Third International Conference on Hot Isostatic Pressing - HIP '91, Theory and Applications. Elsevier Appl. Sci. Publ., Amsterdam.

Hayashi, R., Kawamura, Y., Nakasa, T. and Okinaka, O. 1989. Application of high pressure to food processing: Pressurization of egg white and yolk, and properties of gel formed. Agric. Biol. Chem. 53 (11): 2935.

Horie, Y., Kimura, K., Ida, M., Yosida, Y. and Ohki, K. 1990. Jam preparation by pressurization. Nippon Nogeikagaku Kaishi, 65: 70, 975

Knorr, D. 1992. Effects of high-hydrostatic-pressure processes on food safety and quality. Proc. of the IFT Food Engineering Division Symposium Use of Hydrostatic Pressure in Food Processing. New Orleans, LA, USA.

Marini, D. and Balestrieri, F. 1994. Variability of some analytical characteristics of orange juices. Ital. J. Food Sci. 6:225.

Mertens, B. and Deplace, G. 1992. Engineering aspects of high-pressure technology in the food industry. Proc. of the IFT Food Engineering Division Symposium Use of Hydrostatic Pressure in Food Processing. New Orleans, LA, USA.

Ogawa, H., Fukuhisa, K., Fukumoto, H., Hori, K. and Hayashi, R. 1989. Effects of hydrostatic pressure on sterilization and preservation of freshly-squeezed, non-pasteurized citrus juices, Nippon Nogeikagaku Kaishi 63: 1109.

Rovere, P.P., Carpi, G., Maggi, A. and Dall'Aglio, G. 1993. Produzione e stabilizzazione di purea di mela mediante trattamenti ad alta pressione. Atti CISETA 1° Congresso Italiano di Scienza e Tecnologia degli Alimenti. Parma, Italy.

Saccani, G., Gherardi, S., Trifirò, A., Soresi Bordini, C., Calza, M. and Freddi, M. 1995. Use of ion chromatography for measurement of organic acids in fruit. J. of Chomatogr. 706:395.

Shigehisa, T., Ohmori, T., Saito, A., Taji, S. and Hayashi, R. 1991. Effects of high hydrostatic pressure on characteristics of pork slurries and inactivation of microorganisms associated with meat and meat products. Int. J. of Food Microbiol., 12: 207.

Taka, Y., Awao, T., Asada, K. and Tsujimoto, S. 1990. Sterilization of bacillus sp. spores by hydrostatic pressure. In "Pressure-Processed Food Research and Development". R. Hayashi (Ed.). p. 143 Sanei Publ. Co. Ltd., Kyoto.

Takahashi, Y., Ohta, H., Yonei, H. and Ifuku, Y. 1993. Microbicidal effect of hydrostatic pressure on satsuma mandarin juice. Int. J. of Food Sci. and Technol. 28: 95.

Traff, A. and Bergman, C. 1992. High pressure equipment for food processing. In "High Pressure and Biotechnology". C. Balny, R. Hayashi, K. Heremans and P. Masson (Eds.). vol. 224, p. 509 J. Libbey Eurotext Ltd.

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