INFRA-RED STERILIZATION OF GLASS PACKAGES FOR ASEPTIC PROCESSING

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A pilot plant equipment for continuous dry-heat sterilization of glass containers was constructed and tested.

Containers were heated along a conveyor belt by short-wave infra-red (IR) radiation to a temperature of about 250°C and were then cooled by sterile-filtered air to a temperature of 70-120°C. The input power of the heating stage was 36 kW.

Glass bottles with the volume of 500 ml and the weight of 250 g and 360 g, respectively, were infected on the inside with about 5×10^7 Bacillus subtilis var. niger spores per bottle, heat treated and tested for sterility.

The light bottles were found to be sterile after a heating time of 70 s followed by a cooling time of 290 s. The heavy bottles were sterilized after 80 s of heating and 340 s of cooling. The sterilizing capacity of the pilot plant was 490 light bottles or 430 heavy bottles per h.

The results indicate that the technical obstacles for continuous IR-sterilization of glass containers for aseptic processing can be controlled, and that the sterilization technique can be used for industrial in-line sterilization.

STERILISATION INFRAROUGE DE RECIPIENTS DE VERRE POUR TRAITEMENT ASEPTIQUE

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Un équipement d'installation pilote pour la stérilisation continue par chauffage à sec de récipients de verre a été mis au point et soumis à des essais.

Des récipients ont été chauffés sur bande transporteuse par radiations infrarouges (IR) à ondes courtes jusqu'à une température de quelque 250°C et ont ensuite été ramenés à l'aide d'air stérilisé par filtrage à une température de 70 à 120°C. La puissance d'entrée de la phase de chauffage était de 36 kW.

Des flacons d'une contenance de 500 ml et d'un poids respectif de 250 g et 360 g ont été infectés à l'intérieur à raison d'environ 5×10^7 spores de Bacillus subtilis var. niger par flacon et ont ensuite été traités à chaud et soumis à des essais de stérilité.

Il a été constaté que les flacons légers étaient stériles après un temps de chauffage de 70 s et un temps de refroidissement de 290 s et les flacons lourds après un temps de chauffage de 80 s et un temps de refroidissement de 340 s. La capacité horaire de stérilisation de cette installation pilote atteignait 490 flacons légers et 430 flacons lourds.

Ces résultats ont révélé qu'il était possible de surmonter les obstacles techniques à la stérilisation IR continue de récipients de verre pour traitement aseptique et d'utiliser le procédé ici décrit pour une stérilisation industrielle en série.

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INFRAROTE STERILISIERUNG VON GLASBEHÄLTERN FÜR ANTISEPTISCHE PROZESSE

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Eine Versuchsanlage zur kontinuierlichen Trockenwärme-Sterilisierung von Glasbehältern wurde hergestellt und getestet.

Die Behälter wurden auf einem Förderband durch kurzwellige infrarote Strahlung (IR) auf eine Temperatur von rund 250°C erhitzt und danach durch steril-gefilterte Luft auf eine Temperatur von 70-120°C abgekühlt. Die Eingangsleistung der Erhitzungsstufe betrug 36 kW.

Glasflaschen mit einem Volumen von 500 ml und einem Gewicht von 250 g bzw. 360 g wurden auf der Innenseite mit rund 5×10^7 Bacillus subtilis var. niger Sporen pro Flasche erhitzt und auf Sterilität getestet.

Man fand, dass die leichten Flaschen nach einer Erhitzungszeit von 70 s, gefolgt von einer Abkühlungszeit von 290 s steril waren. Die schweren Flaschen waren nach einer Erhitzungszeit von 80 s und einer Abkühlungszeit von von 340 s sterilisiert. Die Sterilisierungskapazität der Versuchsanlage betrug 490 leichte Flaschen oder 430 schwere Flaschen pro Stunde.

Die Ergebnisse lassen erkennen, dass die technischen Hindernisse einer kontinuierlichen IR-Sterilisierung für antiseptische Prozesse überwunden werden können, und dass diese Sterilisierungstechnik für industrielle Fliessband-Sterilisierung angewendet werden kann.

СТЕРИЛИЗАЦИЯ ИНФРАКРАСНЫМИ ЛУЧАМИ СТЕКЛЯННОЙ ТАРЫ ДЛЯ АСЕПТИЧЕСКОЙ ОБРАБОТКИ

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Была разработана и испытана экспериментальная установка для непрерывной сухой тепловой стерилизации для стеклянной тары.

Тара на конвейерной ленте нагревалась коротковолновым инфракрасным излучением при температуре около 250° С и затем охлаждалась воздухом, простерилизованным посредством фильтрации до температуры 70-120° С. Входная мощность нагревательной ступени составляла 36 кВт.

Стеклянные бутылки емкостью 500 мл и массой 250 г и 360 г, соответственно, инфицировались внутри, приблизительно 5×10^7 Bacillus subtilis или спорами niger /на одну бутылку/, подвергались тепловой обработке и испытывались на стерильность.

Было обнаружено, что стерильность легких бутылок достигалась после операции подогрева в течение 70 сек с последующим охлаждением в течение 290 сек. Для тяжелых бутылок данные показатели составляли 80 сек и 340 сек соответственно. Стерилизующая производительность установки составляла 490 легких бутылки или 430 тяжелых бутылок в час.

Результаты показывают, что технические трудности процесса непрерывной стерилизации инфракрасными лучами стеклянной тары для асептической обработки могут быть устранены, а также подтверждают возможность использования данного метода стерилизации в промышленном поточном производстве.

INFRARED STERILIZATION OF GLASS PACKAGING FOR ASEPTIC PROCESSING

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INTRODUCTION

High-temperature-short-time (HTST) sterilization combined with aseptic filling is a technique which has been used for some time for the processing of sterile milk (Bloomberg and Hessey, 1951, Meaklim, 1964 and Aggarwal, 1974). The technique has also been used for sterilizating other liquids, such as fruit pie filling, puddings, egg nog, custard and liquid coffee whitener (Brody, 1973a and Kent, 1976). Furthermore, the development of a new heating technology has made it possible to apply the HTST-technique to the processing of heterogenous food products. Mathematical models for evaluating the thermal effects of aseptically processing foods containing particulates are already available (Manson and Cullen, 1974).

A drawback of the HTST-aseptic filling technique is the difficulties in providing the filling line with sterilie containers, i.e. it is difficult to design continuous sterilization systems for the packaging material.

Heat sensitive packaging material such as paper-board-foil-plastic is usually sterilized by using hydrogen peroxide combined with a weak heat treatment (Brody, 1973 b, Toledo et al., 1973 and Mann, 1975).

Thermal in-line sterilization of containers has principally been limited to metal cans (superheated steam) and according to Criswell (1975) no commercially proven and generally available system for processing glass containers is to be found. This is in spite of the fact that several systems for continuously sterilizating of glass bottles have been suggested (Dearden and Warrington, 1966, Mann, 1966, Pavey, 1967 and N.N., 1968). Most of the systems used steam as the sterilizing agent.

Heating with infra-red (IR) radiation has been indicated to be a suitable method for continuous sterilizating of glassware (Michel and Sjapilskij, 1974, Molin and Östlund, 1975, 1976 and Molin and Molin, 1976). Molin and Molin (1976) showed, for example, that 10 ml vials could be dry-heat sterilized in 30 s using hig-intensity short-wave IR-radiation.

The aim of the present work has been to elucidate the technical possibilities of using short-wave IR-radiation for the continuous dry-heat sterilization of glassware by developing a pilot-plant equipment and testing its sterilization efficiency. Glass bottles of a volume of 500 ml were chosen as test objects.

MATERIAL AND METHODS

Pilot-plant

Pilot-plant equipment for continuously sterilizating glass bottles was constructed according to our specifications (Infrarödteknik AB, Vänersborg, Sweden). The equipment is shown in figures 1 and 2.

The apparatus consisted of a heating stage and a cooling stage. The heating was performed by short-wave IR-radiation emitted by 34 Philips® LP 360 IR-heaters (each with the input power of 1 kW) compactly arranged along a 0.7 m long tunnel. The bottles were radiated from the sides (by 2 x 12 kW), the top (6 kW) and the bottom (4 kW) standing in file (adjoining each other) on a conveyor belt (lattice of stainless steel).

The cooling was performed by blowing sterile filtered air (temperature about 30°C) over the bottles. The air was distributed over an area of 0.6 x 1.9 m (air velocity; about 1 m/s) by a fan with an input power of 0.8 kW.

The total length of the line (heating and cooling stage) was about three m.

Temperature recording

The temperature of each bottle was, at the end of the heating stage, recorded by a pyrometer (Thermalert® LC 814, Raytek Inc., California) and registered by a Norma® 2200 GB 1 D/a recorder (Norma Messtechnik, Wien). The temperature was measured in the centre of the bottles side in an area of about 18 mm^2.

Glass material

Glass bottles (infusion bottles) with a volume of 500 ml and a weight of 250 g and 360 g, were used in the experiments. According to the manufacturer (PLM - AB Plåtmanufaktur, Malmö, Sweden) the glass contained (% w/w): SO_2, 72.4; Al_2O_3, 1.5; Fe_2O_3, 0.09; MgO, 2.0; CaO, 9.9; Na_2O, 13.4; H_2O, 0.5 and SO_3, 0.2.

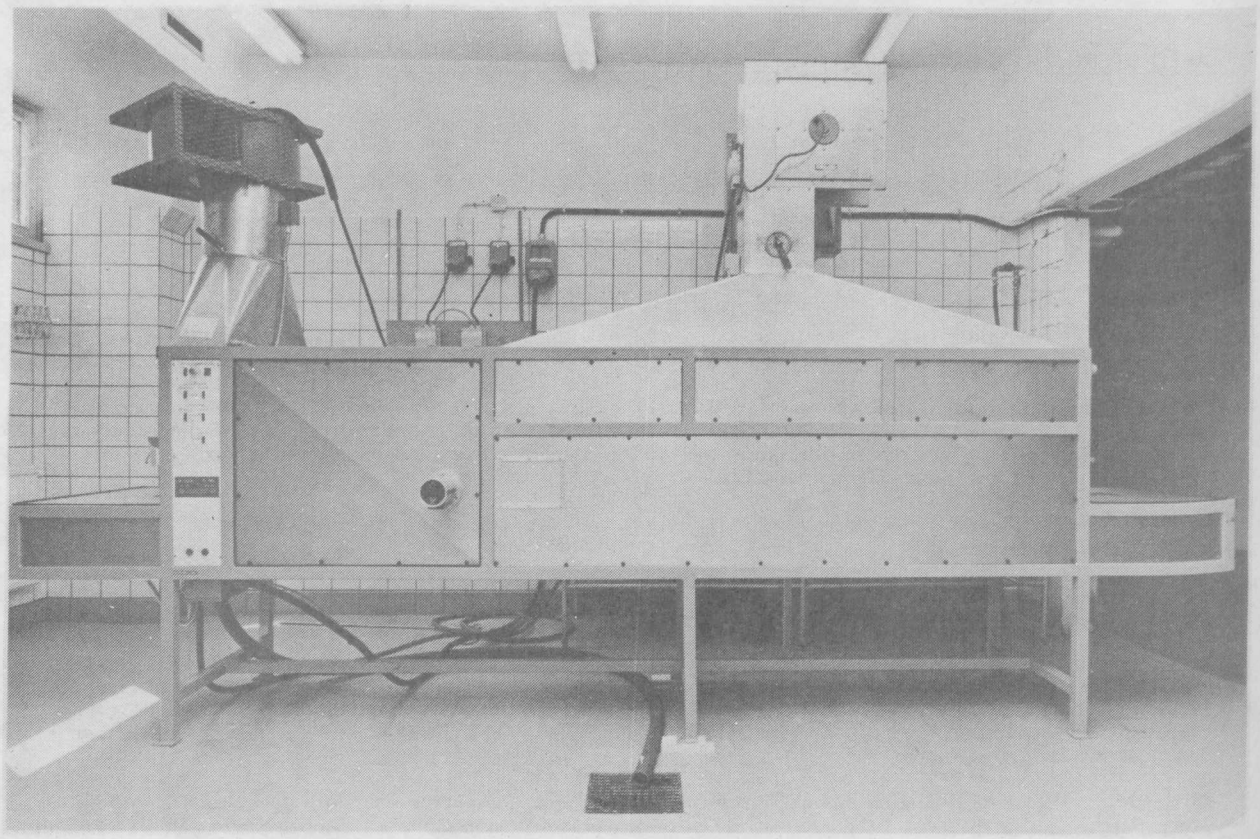

Figure 1. Pilot-plant equipment for the continuous dry-heat sterilization of glass containers.

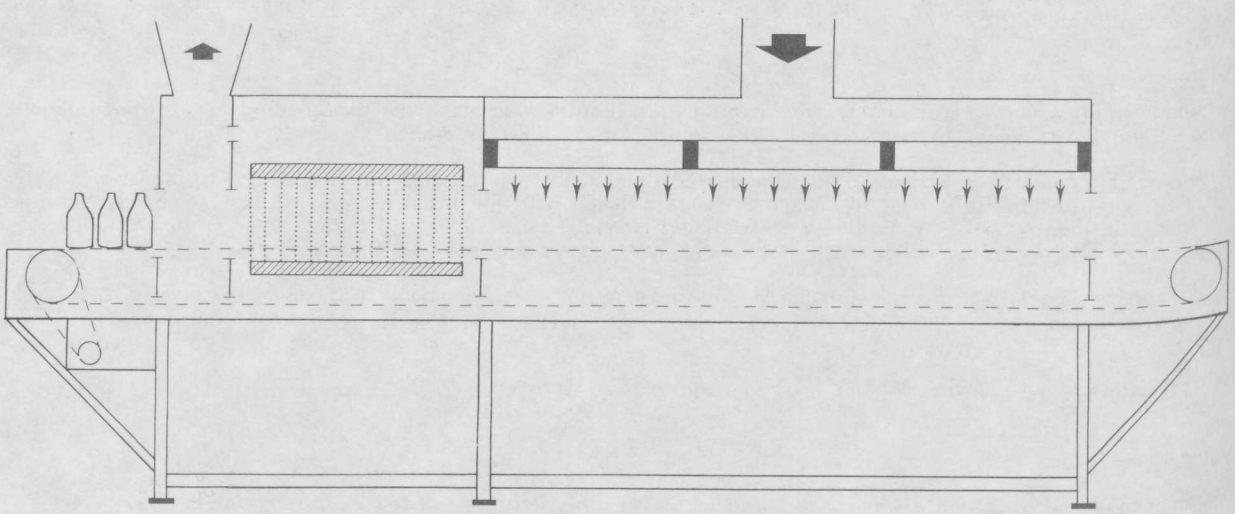

Figure 2. Diagram of the pilot-plant equipment.

Spore material

Bacillus subtilis var. niger ATCC 9372 spores were produced on a solid medium earlier described by Molin and Svensson (1976). After 48 h of incubation at 37°C the spores were harvested, washed and stored in 95% ethanol (Molin and Östlund, 1976).

Glass bottles were inoculated with about 5×10^7 spores per unit by distributing 0.4 ml of spore suspension on the inside of each bottle. The diluent evapurated as the bottles were slowly rotated and the spores were left distributed over the sides and the bottom. The infected bottles were stored in the laboratory for 2-5 days before heat treatment (temperature; 20-24°C, relative humidity; 35-55%).

In order to define the heat resistence of the spore material, spores were applied to glass plates (10^8 spore/sample) and dry-heat inactivated in an open system (Molin and Östlund, 1975) at 150°C, 160°C and 170°C. The D_{150}, D_{160}, D_{170} and the z-values were calculated (Molin and Östlund, 1976).

Heat treatment

The inoculated bottles were heat treated in the pilot-plant to tempeatures of 200-260°C and then cooled to temperatures in the range of 100-150°C. The bottles were treated in series of 10-50 units at a time.

Testing sterility

Heat treated bottles were filled with about 250 ml Tryptone-soya-broth (Oxoid) under laminar air flow conditions and stored for 7 days at 30°C before being examined for growth.

RESULTS

The B. subtilis var. niger spores used as an indicator organism were found to have a D_{150}-value; 370 s (240-820 s), D_{160}; 150 s (100-200 s) and D_{170}; 60 s (50-80 s) when heat inactivated in a dry and open system. Figures within parenthesis indicate the 95% confidence limits. The z-value was found to be 25°C.

Bottles inoculated with B. subtilis var. niger spores were heat treated in the pilot-plant and then tested for sterility. See table 1.

Table

Infra-red heating of 500 ml bottles inoculated with 5×10^7 Bacillus Subtilis var. niger spores per unit.

Type of bottles	Heating time (s)	Endpoint temp. (°C)	Cooling time (min.)	Capacity (units/h)	Number of sterile units/ /Number of tested units
Heavier	90	247	4.2	400	10/10
	90	252	4.2	400	20/20
	90	247	4.2	400	20/20
	80	241	3.8	440	10/10
	75	226	3.5	480	10/10
	75	232	3.5	480	10/10
	75	230	3.5	480	20/20
	70	217	3.2	500	5/10
	60	199	2.9	570	0/10
Lighter	80	254	3.6	460	10/10
	80	252	3.6	460	20/20
	80	257	3.6	460	19/20
	80	254	3.6	460	19/20
	70	247	3.2	500	8/10
	65	234	3.1	530	10/10
	60	220	2.7	600	9/10
	55	217	2.5	650	10/10
	55	217	2.5	650	20/20
	55	219	2.5	650	20/20
	50	209	2.3	710	0/10
Control x)	90	250	4.2	400	48/50
	90	250	4.2	400	49/50

x) Heavier bottles sterilized in 160°C for 3 h before treatment.

Bottles reaching the temperature of 220-230°C were found to be sterile. The time needed to sterilize the heavier bottles was somewhat longer than that needed for the lighter bottles. Thus, the light bottles were sterilized after a heating time of 55 s (cooling time; 2.5 min.) compared with a heating time of 75 s for the heavier bottles (cooling time; 3.5 min.). The sterilization capacity of the pilot-plant was about 650 of the lighter bottles per h and 480 of the heavier bottles per h.

The end-point temperatures shown in the table refer to the mean value of the whole series of treated bottles. The maximum deviation, from the mean value, was 9°.

No damage to the glass could be observed in any of the bottles heated to the sterilization temperatures (250 units).

DISCUSSION

It was shown in the present study that bottles can be heated by short-wave IR-radiation to 220-260°C for 60-90 s without breaking the glass. Furthermore, it was indicated that this treatment was adequate in sterilizing the bottles. Thus, when the bottles were infected with 5×10^7 B. subtilis spores per unit and heated to the critical temperatures, totally 245 units out of 250 tested were found to be sterile (98%).

The appearence of unsterile units probably results from recontamination during cooling or during sterility testing of the bottles. This is supported by the fact that when 100 sterile bottles (sterilized in a hot air oven at 160°C for 3 h) were treated in the pilot-plant, only 97% of the bottles were found to be sterile according to the sterility testing.

The dry-heat resistance of the spores used as the indicator organism in the present study was determined in the temperature range of 150-170°C. According to earlier experiments (Molin and Östlund, 1976 and Molin, 1976), the z-value is constant over a wide temperature range in this type of heating system. The inactivation data found in the present study may therefore be extrapolated to other inactivation temperatures with reasonable accuracy. Thus, for example, the D_{195}, D_{220} and D_{245}-value would be about 6 s, 0.6 s and 0.06 s, respectively. Consequently, the time needed to inactivate 10^7 spores at 220°C would be about 4 s (excluding the heating up and cooling down periods) which corresponds reasonably well with the sterilization results found for the bottles in the present investigation.

B. subtilis var. niger, which was the only test organism used in this study, is known to be a suitable indicator organism for dry-heat sterilization processes (Craven et al., 1968 and Costin and Grigo, 1974). Furthermore, it has been shown earlier that (i) B. subtilis var. niger (ATCC 9372) was the most dry-heat resistant organism out of 9 different Bacillus organisms tested (Molin, 1976) and that (ii) the sporulation medium used was the most suitable for producting dry-heat resistant B. subtilis var. niger spores out of 20 different media (Molin and Svensson, 1976).

However, naturally occurring spores are generally considered to have higher heat resistance than those formed on a laboratory medium (Bond et al., 1970, and Puleo et al., 1975). Bond et al. (1970) showed that the dry-heat resistance, in terms of D_{125}-value, of spores collected from soil varied in the range of 11-38 min and Puleo et al. (1975) showed that spores collected from "Manned Spacecraft Operation Building" varied in the range of 25-126 min. Nevertheless, the extrapolated D_{125}-value, of the present investigation, is about 60 min. Thus, the dry-heat resistance of the spore material used seems to correspond fairly well to that of naturally occurring spore populations. Considering this, it is suggested that the sterilization cycles reported here are valid for short-wave IR-heating in dry and open systems. Even so, more extensive tests are required before the system can be used in practice.

The present investigation has confirmed earlier suggestions that short-wave IR-radiation is suitable for continuous heat sterilization of glassware (for example; Molin and Östlund, 1975 and Molin and Östlund, 1976). Compared with other tested principles for continuously heating glass containers, i.e. heating with steam (Pavey, 1976, and N.N., 1968) it seems that heating with short-wave IR-radiation has the advantage of being technically more simple and verifiable and is also cheaper from the investment point of view. Thus, the heating unit is small and compact and provides simple installing arrangements making it easy to design heating lines for a wide range of different applications with different demands on heating capacity.

No indications were found that IR-heating should cause any mechanical stresses within the glass and it was indicated that the heating rate could be increased further without causing damage. However, the cooling of the hot bottles must be carried out with care so as to avoid big temperature gradients that may cause the glass to break. In order to make the cooling more efficient it is suggested that the velocity of the cooling air is successively increased along the tunnel or that the air temperature is relatively high at the beginning of the tunnel and then is decreased along the cooling stage. It ought to be noted that the cooling stage of the present apparatus was designed in a relatively primitive way and that the cooling efficiency may thus be considerably improved.

In concluding it may be said that the presented pilot-plant is not a completed equipment but that the obtained results proved that the principle of short-wave IR-heatin could be used for short-time sterilization of glass containers.

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