

Aseptic Packaging of Liquid Food Products: A Literature Review

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"With 25 billion units annually produced worldwide at a growth rate of about 10%, the art of packing a sterile product in a sterile container in a sterile environment—i.e., aseptic packaging—appears to offer the best of all possible worlds. It gives portable liquids long bacteria-free shelf life while helping retain their nutritional integrity. It also makes possible the reduction of material costs by 25-30% compared to tin plate or glass, is biodegradable, and has a high-energy recovery factor" (Beverage World, 1983). Aseptic technology permits use of thermoplastic and paper-based materials in applications previously confined to metal or glass. It provides inexpensive packaging in small sizes, virtually creating a single service market. Aseptic containers are biodegradable, incinerable, and recyclable. They can withstand long shipping and storage and take up less space.

A. INTRODUCTION

Commercially sterile products have been on the market for a long period of time. The usage of such products can be seen in three different market sectors (Farahnik, 1982): direct consumption and private households, many rigid and flexible packages having a size of up to ca. 1 L; institutional market, rigid containers and bag-in-box in sizes of up to about 5 L; industrial market, drums and tanks in the size range of 100-1000 L.

In order to achieve commercial sterility, both product and container have to be sterilized. Basically this can be done in two different ways: sterilization of the product and container together, the retort or autoclave procedure; sterilization of product and container separately, the aseptic technology.

In this paper, only aseptic packaging procedures in flexible or semirigid packaging materials will be dealt with. Further, main emphasis will be given to its use for "direct consumption", i.e. to packaging systems producing containers of up to about 1 L of volume. Flexible and semirigid materials will include all materials used commercially with the exception of glass and metal. There is a worldwide trend to replace the metal can by other kinds of containers (*Verpackungs-Rundschau*, 1983). Today, three types of aseptic packaging systems are most common: flexible, plastic pouches or bags, thermoformed or deep-drawn plastic cups, and various types of paper-based laminates (Kelsey, 1974a).

In a draft on "Recommended Canadian Code of Practice for Low-Acid and Acidified Low-Acid Canned Foods" (Canadian Health Protection Department, Ottawa, Canada) the following definitions are given:

Rigid means that the shape or contours of a filled and sealed container are neither affected by the enclosed product nor deformed by an external mechanical pressure of up to 70 kPa.

Semirigid means that the shape or contours of a filled, sealed container are not affected by the enclosed product under normal atmospheric temperature and pressure but can be deformed by an external pressure of less than 70 kPa.

Flexible means that the shape of contours of a filled, sealed container are affected by the enclosed product.

B. GENERAL PROCEDURES

Aseptic packaging has been defined as sterilization of the packaging material or container, filling of a commercially sterile product in a sterile environment, and producing containers that are tight enough to prevent reinfection, i.e., that are hermetically sealed (B. von Bockelmann, 1970, 1978b; Farahnik, 1982; Hallström, 1979; Food and Drug Administration, 1980). The term "aseptic" implies the absence or exclusion of any unwanted organisms from the product, package, or other specific areas (Carlson, 1980), while the term "hermetic" is used to indicate suitable mechanical properties to exclude the entrance of bacteria into a package or more stringently to prevent the passage of microorganisms and gas or water vapor into or from the package (Dickerson and Read, 1972; Food and Drug Administration, 1980).

The performance level of an aseptic plant is controversial (B. von Bockelmann, 1985a). Obviously, aseptic packaging procedures should maintain the high level of microbiological quality of the sterilized product, i.e. the state of commercial sterility. Unsterile packages in an aseptic operation, however, originate not only from the packaging operation but also from the product sterilization process as well as from any reinfection of the properly sterilized product on its way from sterilizer to packaging machine (I. von Bockelmann, 1985). The performance level of an aseptic plant must be looked upon as a performance level of the entire process rather than one of a single component of the production line (Cerf and Brissenden, 1981).

The state of commercial sterility has been defined as "... the absence of microorganisms capable of reproducing in the food under normal nonrefrigerated conditions of storage and distribution and ..." (Food and Drug Administration, 1980). However, the absolute absence of all microorganisms cannot be achieved (Heim and Jud, 1973). Statements like "the destruction of all microorganisms" should be avoided (Cerf and Brissende, 1981). The logarithmic order of death makes it impossible to destroy all microorganisms (Cerny, 1978; Kelsey, 1974b). Consequently, "maximal acceptable defective rates" have been discussed for this technology. A "maximal acceptable defective rate" of 1:1000 (0.1%) has been suggested

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(*Deutsche Molkerei-Zeitung*, 1982; Cerf and Brissenden, 1981; Rippen, 1970; Samuelsson and Kristiansen, 1970; Swartling and Lindgren, 1962). The general rejection rate of aseptically packaged products was estimated to be 1:10000 (0.01%) (Lembke, 1972), and even a "sterile guarantee" of 0.01% is offered for one aseptic packaging system (Lütkemeyer, 1983). The actual defective rate is determined by the sterilization efficiency of the product sterilizer, the microbial load of the raw material fed into the sterilizer, cleaning and plant-sterilization procedures applied, sterilization efficiency of the packaging material sterilization process, microbial load on the packaging material food contact surface, cleaning and filler sterilization procedure, and service and maintenance of the plant as well as operational care (I. von Bockelmann, 1985).

With regard to the sterilization of the container food contact surface, the following equation has been developed to calculate the risk of defectives originating from this source (Cerf and Brissende, 1981):

$$R = C_0 S \times 10^{-t/D}$$

where R = risk, C_0 = number of the most resistant organisms per square centimeter, S = square centimeter of the food contact area, t = time of the sterilization process, and D = decimal reduction time of the most resistant organisms.

Consequently, the risk is strictly proportional to the initial contamination level and the area of the unit container and depends on the efficiency of the lethal treatment.

Prior to the start of an aseptic packaging operation, the filler must be sterilized. Sterilization of the packaging equipment involves surface sterilization procedures applying heat, chemicals, or both. Time might be necessary to reach temperature equilibrium, but once such an equilibrium is reached, the sterilization process can be described by the formula (Toledo, 1978)

$$\log(N_0/N) = \text{contact time with sterilant}/D$$

where N_0 = initial number of viable organisms, N = viable count after a given time of contact with the sterilant, and D = decimal reduction time of the organisms.

In aseptic packaging machines, valves, pipes, etc., are usually sterilized by live steam at temperatures of 120–140 °C (*Deutsche Milchwirtschaft*, 1973; Buchner, 1980; Linke, 1971; Voss, 1974; Zimmermann, 1974). Chambers, tunnels, cabinets, etc., are often sterilized by a spray of a suitable disinfectant, usually hydrogen peroxide, and subsequently dried with hot sterile air (*Deutsche Milchwirtschaft*, 1974a; Buchner, 1980; Kelsey, 1974b; Toledo and Chapman, 1973; Voss, 1974).

Since commercially sterile products have an extended shelf life at ambient temperatures, the following requirements have been listed for the packaging material and/or containers (Ashton, 1972): to be an effective barrier to the transmission of light; to be impermeable to gases and vapors; to resist absorption of moisture; to be neutral as far as flavor or taints of the packaged product are concerned; to be free from substances that are toxic or harmful to health; to resist any chemical and/or heat treatment applied preparatory to filling; to be capable of being hermetically sealed; to resist deteriorative changes during storage; to conform to specifications; to give minimum waste during preparation; to be easily disposable; to be light in weight and relatively inexpensive; and to meet general consumer acceptance.

Of primary interest and concern is the package strength and integrity (Carlson, 1980), as well as its performance with regard to pressure differences (air transportation, different ground levels, etc.), changes in temperature of storage, behavior during transportation, etc.

Practical solutions will always be a compromise based on the above list of desirable characteristics. No single, individual packaging material, package, container, or packaging system can possibly be optimal in all aspects listed.

C. MICROBIOLOGICAL ASPECTS

In the production of shelf-stable, aseptically packaged food products, usually four separate processes of sterilization are involved: sterilization of the process equipment; sterilization of the product; sterilization of the filler; sterilization of the packaging material. In addition, often sterile air is required in the aseptic filling operation necessitating an air-sterilization procedure.

Sterilization processes are aimed at the killing and/or elimination of all microorganisms present. Applying chemical means or heat treatment, the rate of killing in large follows a logarithmic order. Consequently, absolute sterility can only be approached, but never reached (Kelsey, 1974a,b). This, of course, is also valid for the sterilization of food contact surfaces. With regard to flexible or semirigid packaging materials, the suitability of sterilization by heat alone has been questioned (Kelsey, 1974b; Voss, 1974), and other means of sterilization should be looked for, such as irradiation or chemical sterilization. In general, the following characteristics must be fulfilled by such a sterilization procedure (Toledo, 1975): sporicidal activity; applicability in aseptic packaging systems; compatibility with packaging material; ease of removal from the treated food contact surface; tolerance for residues (lack of toxicity).

A perfect sterilant should be easy to apply, part of an in-line operation, and residue free after application. It must have a sufficient kill rate in the shortest possible time, it should be inexpensive, and it should not be toxic to the user nor damage the equipment (Farahnik, 1982).

Prior to a discussion of the killing efficiencies necessary in the process of sterilization of packaging material food contact surfaces, consideration should be paid to the microbial load present on such surfaces. Among other factors, the microbial load of the packaging material food contact surface as well as the sterilizing effect of the packaging material sterilization system are related to the defective rate of such an operation (*Deutsche Molkerei-Zeitung*, 1982).

Average microbial counts determined on plastic food contact surfaces range from 0.3 to 10 microorganisms/100 cm² (B. von Bockelmann, 1970, 1971, 1978a, 1980; Brody, 1971; Buchner, 1978a; Cerny, 1982; Heim and Jud, 1973; Lisiecki, 1971; Lubieniecki-von Schelhorn, 1970; Swartling and Lindgren, 1962; Voss, 1974). Though the total microbial load on food contact surfaces to be sterilized is of some interest, the kind of microorganisms and their counts are even more so. Very few data are available in this respect, and all originate from the same source (B. von Bockelmann, 1970, 1978a, 1980). The following microflora were found on polyethylene food contact surfaces of paperboard-based laminates: average total count 2–5 microorganisms/100 cm², with 10.6% yeast, 20.6% molds, and 68.8% bacteria. A further differentiation of the bacterial flora showed (based on the total microbial count) the following: 44.4% *Micrococci*, 3.1% bacterial spores

(*Bacillus*), 3.7% *Streptococci*, 1.2% *Pseudomonas*, 6.9% Gram-positive rods, and 9.4% Gram-negative rods.

All the above results were obtained immediately after producing the packaging material, not accounting for further infection of the food contact surface at the site of the commercial processor where the filling equipment is operated. The microbiological data on food contact surfaces clearly indicate an air-borne infection. Nothing is published about changes in number and composition of these flora during storage of the packaging material. It seems unlikely that Gram-negative rods (*Pseudomonas*) and yeasts will survive longer periods of time. Considering the goal of aseptic packaging, the bacterial spore count appears to be of predominant interest since these are the microorganisms most difficult to eliminate (Toledo et al., 1973).

The efficiency of a sterilization process can be expressed by the number of decimal reductions in spore counts achieved. If highest acceptable failure rates are decided upon, minimal decimal reduction values can be calculated. Maximal acceptable defective rates in aseptic food production plants seem to vary between 1:1000 (*Deutsche Molkerei-Zeitung*, 1982; Swartling and Lindgren, 1962) and 1:10 000 (Buchner, 1985; Lembke, 1972; Lütkemeyer, 1983). The failure rate of a sterilization process is mainly determined by the number of the most resistant microorganisms present—i.e. bacterial spores—and not by the total count. Little is known about spore counts on packaging material food contact surfaces. Consequently, calculations of minimum sterilization effects (decimal reductions) necessary are often based on the assumption that all microorganisms on food contact surfaces are bacterial spores. Sterilization of such surfaces has been defined as a reduction of the microbial load from 10^4 to 10^0 , i.e. four decimal reduction cycles (Lubieniecki-von Schelhorn, 1970). For aseptic packaging of milk and milk products, procedures should be chosen that reduce the number of spoilage organisms by four to six decimals (Gruber and Ziembra, 1970; Hahn, 1981). Four to five decimal reductions are considered necessary to obtain a maximal spoilage rate of 5 in 10 000 (Heim and Jud, 1973). Defining a sterilization process by killing rates against bacterial spores appears to be a more suitable way. According to Lütkemeyer (1983), four decimal reductions in spore counts are required in the U.S. as a minimum for the sterilization of food contact surfaces. This opinion is shared by others (B. von Bockelmann, 1970; Cerny, 1985a,b; Swartling and Lindgren, 1962). In order to meet the requirements of aseptic filling of neutral food products, five to six decimal reductions of bacterial spore counts are regarded to be necessary (Cerny, 1982).

D. STERILIZATION OF PACKAGING MATERIAL FOOD CONTACT SURFACES

Sterilization of packaging material food contact surfaces requires a minimum of four decimal reductions against bacterial spores (B. von Bockelmann, 1970; Cerny, 1985b; Hahn, 1981; Lubieniecki-von Schelhorn, 1970; Lütkemeyer, 1983; Swartling and Lindgren, 1962).

Sterilization of packaging material food contact surfaces can be achieved by different means or by combinations thereof (B. von Bockelmann, 1985a): irradiation, heat, chemical treatment.

a. Irradiation. Particle irradiation techniques using cobalt 60 and the like are regarded as too expensive (Kelsey, 1974a,b). A sterilizing procedure using high-en-

ergy electron irradiation has been described (Modern Packaging, 1972). A 2-Mrd dose of high-velocity electrons was found to sterilize 0.353 in. of a polyethylene strip infected with ca. 10^5 spores of *Bacillus stearothermophilus* (Mayernik and Daniels, 1959).

The advantages of low-energy (100 keV), large-area electron beams for the surface sterilization of packaging/container materials have been pointed out (Nablos and Hipple, 1972).

Ultraviolet irradiation has been developed further in recent years (Mayer, 1975, 1976a,b). Statements on the efficiency of the sterilization of food contact surfaces vary. Good killing efficiencies are reported by some researchers. A dose of 30 mW/cm² is stated to result in four decimal reductions in the count of *Bacillus subtilis* spores after 0.3 s of exposure while fungal spores required 1 s of exposure time to achieve the same killing effect (Cerny, 1982). A few seconds of exposure were needed to accomplish four to six decimal reductions in microbial counts on flat surfaces when using a dose of 250 mW/cm² (*Verpackungs-Rundschau*, 1981). For sterilization of preformed cups, about 5 times this dose was required (*Verpackungs-Rundschau*, 1981). UV-C irradiation (Brown Beverly) is regarded to be sufficient for use in aseptic filling systems provided that the irradiated materials are smooth, UV-resistant, and rather free from dust particles (Cerny, 1977). Dust as well as "shadows" interfere with UV sterilization procedures (*Verpackungs-Rundschau*, 1981; Cerny, 1978).

Only 90–99% reduction of the microbial load on plastic cups is reported after UV irradiation (up to 1500 mW/cm²), the limited sterilizing effect being explained by dust and microbial cell aggregation (*Gasti Informiert*, 1980). With *Aspergillus niger* as test organism, only two decimal reductions resulted after UV-C irradiation (*Deutsche Molkerei-Zeitung*, 1982).

Though UV irradiation alone hardly provides the safety of operation needed when sterilizing food contact surfaces for aseptic packaging of low-acid foods, a combination of relatively low concentrations of hydrogen peroxide and UV irradiation very well might. UV irradiation of *B. subtilis* spores in the presence of hydrogen peroxide produced a rapid kill that was up to 2000 times faster than the one by irradiation alone (Bayliss and Waites, 1979a,b). More resistant strains of *Bacillus* and *Clostridium* required a mild heating—60 s at 80 °C—in order to achieve a minimum of four decimal reductions in spore counts after UV irradiation in a 2.5% solution of hydrogen peroxide (Bayliss and Waites, 1979a,b; Standard et al., 1983). UV irradiation and hydrogen peroxide act synergistically when used together but not when applied separately. Irradiation of spores in the presence of higher concentrations of hydrogen peroxide reduced the kill, which is explained by absorption of the UV light (Bayliss and Waites, 1979b; Standard et al., 1983).

The sporicidal action of hydrogen peroxide is seen in the formation of hydroxyl radicals, which are produced when hydrogen peroxide is irradiated with light of wavelengths below 400 nm (Bayliss and Waites, 1979b).

Under practical conditions of use, the combined application of hydrogen peroxide and UV irradiation was found to be less effective than expected. As compared to UV irradiation alone, only a 5-fold increase in sterilization efficiency was observed when sterilizing erected carton blanks having a polyethylene inner coating. This effect is explained by the hydrophobic characteristics of the polyethylene resulting in only 35–40% coverage after application of hydrogen peroxide by spraying (Cerny, 1985).

b. Heat. Considering characteristics of flexible and

semirigid packaging materials available, the suitability of heat alone as a sterilizing agent has been questioned (Kelsey, 1974a; Voss, 1974). However, by extrapolating published values with regard to the resistance of bacterial spores to dry heat, temperatures of 250 °C for 0.01–0.02 s should be sufficient to kill 10^{10} spores of *B. subtilis* (Brigaud et al., 1970). Using flame heating under laboratory conditions, *B. subtilis* spore counts were reduced in 0.05 s from 10^5 to 0.17×10^{-5} at 280 °C to 0.57×10^{-5} at 250 °C and to 2×10^{-5} at 220 °C (Brigaud et al., 1970).

Manufacturers of aseptic filling machines using plastic pouches (Dairy Industries, 1972; Ashton, 1972; Leali et al., 1970; Uteschill, 1971) or plastic bottles (*Deutsche Milchwirtschaft*, 1974b, 1975b, 1982; Dairy Industries, 1972; *Neue Verpackung*, 1971; Ashton, 1972; Mann, 1975b; Zimmermann, 1974, 1975) claim that the heat of extrusion suffices for sterilization. For blow molding of plastic bottles this temperature is stated to be ca. 230–240 °C (*Neue Verpackung*, 1971; Mann, 1975b; Zimmermann, 1975). The result will depend upon the microbial load of the plastic granulate (Heim and Jud, 1973; Lubieniecki-von Schelhorn, 1970; Voss, 1974), however, may suffice for practical purposes (Voss, 1974).

Superheated or saturated steam—which has been applied for sterilization of cans (Brody, 1971)—could also be used for the sterilization of flexible packaging material food contact surfaces (Hallström, 1979). Using polystyrene cups, saturated steam under high pressure has been applied for this purpose (Cerny, 1982, 1983). Bacterial spore counts were reduced by 5.5 to almost 7 logarithmic cycles when exposed for 4–6 s to saturated steam at a temperature of 147 °C and 3.5 bar overpressure (Cerny, 1983). Using a temperature of 165 °C and saturated steam at 6 bar overpressure, five to six decimal reductions in *B. subtilis* spore counts were registered (Cerny, 1982).

c. Chemical Treatment. Chemical sterilization of food contact surfaces is the most frequently applied procedure in aseptic packaging. A review (Toledo, 1974) covers gaseous as well as liquid sterilants. The action of gases (ethylene oxide, propylene oxide, β -propiolactone, formaldehyde, methyl bromide) is too slow to be used in filling machines. Such gases may, however, be useful in pre-sterilization of the packaging material. Halogen solutions, hydrogen peroxide, peracids, and aldehyde solutions can be used in liquid form. Hypochlorite at a concentration of 4000 ppm had been studied in the early 1960s (Swartling and Lindgren, 1962). Though good sterilizing effects are reported, corrosion problems prevented practical application. Peracetic acid as well as aldehyde solutions have been shown to have sporicidal properties; however, pungent and irritating odor or vapors, off-flavor problems resulting from residuals, and relatively long exposure times required rendered these compounds less suitable (Toledo, 1974).

Hydrogen peroxide appears to be the most promising sterilant. At ambient temperatures hydrogen peroxide possesses very slow sporicidal activity. However, at elevated temperatures the *D* values decrease rapidly in a logarithmic order (Toledo, 1974). Chemical sterilization of food contact surfaces requires fast microbiocidal action, no negative effects on the packaging material, good possibilities for elimination, no corrosion in the filling equipment, and no danger in handling; these demands are met by hydrogen peroxide (Heim and Jud, 1973).

Today, most aseptic packaging systems are using hydrogen peroxide as a sterilant of the food contact surface. Literature reviews are published on the microbiocidal action of hydrogen peroxide (von Bockelmann and von Bockelmann, 1972; Stevenson and Shafer, 1983) covering

the effect of concentration, exposure times, temperature of exposure, and pH.

Hydrogen peroxide is regarded as the most suitable chemical sterilizing agent for treatment of food contact surfaces in aseptic packaging of low-acid foods, provided it is heated to a temperature of at least 85–90 °C and the time of exposure is at least 3–4 s (*Gasti Informiert*, 1980). A 15–20% solution of hydrogen peroxide was found to meet the theoretical and practical requirements if followed by a heating to about 125 °C (Swartling and Lindgren, 1962); the killing effect was explained by a combined action of liquid and gaseous (vapor) hydrogen peroxide. Others (Gruber and Ziemba, 1970; Hahn, 1981) claim that a safe killing rate (at least four decimal reductions) of bacterial spores can only be achieved in seconds if the concentration of the hydrogen peroxide used is at least 30% and the temperature on the food contact surface reaches at least 80–90 °C. Wetting of the surface is not regarded to be essential since hydrogen peroxide in the gas phase is more powerful than as a liquid (Gruber and Ziemba, 1970; Hahn, 1981).

Organic material—provided it is free from catalase—has little effect upon the sporicidal action of hydrogen peroxide (Ito et al., 1973). An increase in temperature and concentration increased the sporicidal efficiency of hydrogen peroxide solutions (O'Connell, 1974; Ito et al., 1973). A hydrogen peroxide concentration of 50% (w/v) is regarded to be optimal. A short-time destruction of the most resistant *Bacillus* spores was only observed at temperatures above 80 °C (Cerny, 1976).

Working in a liquid environment, survivor curves of bacterial spores appear to be variable in shape (Ito et al., 1973). Except for an initial lag period, inactivation curves followed first-order kinetic and an increase in hydrogen peroxide concentration and/or temperature reduced the lag period (Ito et al., 1973). On the other hand, survival curves of *Bacillus* spores after hydrogen peroxide treatment (15%, 80 °C, pH 2.9–7.7) were reported to be non-logarithmic (Cerf and Hermier, 1972), an observation that was explained by lumping of spores (Cerf and Metro, 1977).

After inactivating *B. subtilis* spores with hydrogen peroxide, reactivation could be observed by heating the treated spores (30% hydrogen peroxide, 5 min, 24 °C) to 50–80 °C. Above and below this temperature range no reactivation was observed (Cerny, 1975).

The action mechanism of hydrogen peroxide is still not fully understood (Heim and Jud, 1973). It is attributed to the formation of nascent oxygen (Kelsey, 1974a,b) or to the formation of hydroxyl radicals (Bayliss and Waites, 1979b).

Orthophosphoric acid at a concentration of 80% has also been suggested as a sterilant for food contact surfaces in aseptic packaging (Maile, 1983, 1984). At 30 °C the following *D* values are reported: *B. subtilis*, 6 s; *B. globigii*, 9 s; *B. stearothermophilus*, 57 s. The killing action is explained by complex binding of metals, specifically calcium, affecting the resistance of bacterial spores as well as by the low-pH value.

E. ASEPTIC PACKAGING

a. General Procedures. The history of aseptic packaging shows a development from metal cans to paper-board-based and plastic materials (Brody, 1971; Burton, 1961; Carlson, 1984; Mann, 1984). In large, glass bottles have been rather unsuccessful in the market place as containers for aseptic filling operations. The first aseptic filling procedures introduced on the market used metal

cans. As far as flexible packaging materials are concerned, aseptic filling of food products started with web-fed, paperboard-based packaging materials.

In general, four different ways exist to accomplish aseptic filling (Linke, 1971): (1) use of presterilized packaging material and filling equipment operating in a microbiologically clean area; (2) application of prefabricated, nonsterile packages to be sterilized in the filler; (3) prefabricated, presterilized packages to be filled in a microbiologically clean filling equipment; (4) production, filling, and sterilization of the packages or packaging material in the filler. The first and third alternatives have been and are used in the pharmaceutical industry but are regarded to be too difficult and expensive for the food industry (Buchner, 1978a).

In order to maintain the high level of microbiological quality achieved by UHT (ultrahigh-temperature) processing of various food products, aseptic filling procedures must be applied since "it does not make sense to produce food products with a high hygienical standard if this is spoiled by inadequate packaging procedures" (Cerny, 1978).

Aseptic packaging implies three different steps (B. von Bockelmann, 1970, 1978b, 1985b; Farahnik, 1982; Hallström, 1979): (1) sterilization of the packaging material food contact surface; (2) creating and maintaining a sterile surrounding in the area where the sterilized product and the sterilized packaging material/package are brought together; (3) production of containers that are tight enough to prevent entry of spoilage organisms. As far as flexible and semirigid packaging materials are concerned, the following systems have entered the market of aseptic filling: (1) pouches or bags; (2) prefabricated cups; (3) form-fill-seal cups from roll-stock material; (4) plastic bottles; (5) prefabricated, paper-based laminated cartons; (6) cartons produced from roll-stock material of paper-based laminates.

Unfortunately, practically all literature available on aseptic packaging systems has been published by the manufacturers of such equipment. Very few statements are available on the microbiological performance of these filling procedures. It is, however, safe to assume that systems having successfully entered an area also meet the performance demands valid in that specific market.

b. Aseptic Packaging Systems. 1. Aseptic Pouches. As far as aseptic pouches are concerned, two different procedures are applied for the sterilization of the plastic food contact surface: chemical/physical sterilization in the filler; heat by tubular extrusion outside the filler.

Capacities vary between 2000 and 4000 sachets/h and filling head. Multiple filling head systems are available. Prior to production, the filler cabinet is sterilized by spraying hydrogen peroxide and/or other suitable disinfectants such as formaldehyde (Mann, 1977, 1978) or a germicidal soap (Kelsey, 1974a,b) with subsequent drying by hot, sterile air. The sterility of the system thus achieved is maintained by an overpressure of sterile air fed into the enclosed superstructure of the filler (*Deutsche Milchwirtschaft*, 1974a; Dairy Industries, 1967; Uteschill, 1971). Piping, valves, etc., are usually sterilized by saturated, live steam.

Chemical Sterilization of the Packaging Material Food Contact Surface. The packaging material, usually a multilayered plastic laminate, is supplied in one (Dairy Industries, 1972; Kelsey, 1974b; Lubienicki-von Schelhorn, 1970) or two webs (Ashton, 1972; Mann, 1977, 1978). Sterilization of the packaging material is done by passage through a hydrogen peroxide bath followed by UV irradiation or by "active agents on alcohol basis" (Lubienicki-von Schelhorn, 1970), ethyl alcohol at 95 °C followed by high-intensity UV irradiation (Kelsey, 1974a,b). The sterilization solution is dried off by hot, sterile air (Jentsch, 1970).

Sterilization of the Packaging Material Food Contact Surface by Tubular Extrusion. Heat is applied in the extrusion process, used in the manufacture of the packaging material tube. The temperature of ca. 230–240 °C reached in the process is regarded as sufficient to establish the necessary degree of sterility. The plastic material is extruded in tubular form, the layer being thick enough to prevent entry of any potential spoilage organism from the outside. Consequently, in the filling operation only the infected outside of the material tube has to be sterilized in order to maintain sterility in the filling equipment. Before entering the sterile chamber of the filler, the flat web of tubular packaging material passes through a bath of hydrogen peroxide followed by UV irradiation (Ashton, 1972; Mann, 1977, 1978). A segment is then sealed and cut off the web, opened, filled, and top sealed. The filled and sealed pouches leave the filler through a sterile lock.

In a different approach, a sandwiched plastic packaging material is used (*Neue Verpackung*, 1979; *Verpackungs-Rundschau*, 1980). In the packaging operation the two layers are separated. The outside layers function as barrier against the unsterile surrounding, the product coming into contact with the sterile inner layers only.

2. Aseptic, Prefabricated Cups. Aseptic fillers using prefabricated plastic cups are equipped with a closed superstructure that is sterilized by spraying hydrogen peroxide on all inner surfaces and subsequent drying by hot sterile air. Piping, valves, etc., are usually sterilized by steam. Sterility of the superstructure is maintained by an overpressure of sterilized air. Air sterilization is usually achieved by filtration or incineration.

As far as prefabricated aseptic cups are concerned, four different means of sterilization of the packaging material food contact surface are being used: heat; UV irradiation; chemical sterilization; use of presterilized cups.

Sterilization by Heat (Cerny, 1983). Sterilization of both the cup body and lid material is done by saturated steam at a temperature of 147 °C and a pressure of 3.5 bar. The time required for both heating and holding amounts to 4–6 s. By this treatment, a reduction of five and one-half to seven decimal cycles is reported using *Bacillus* spores as test organism. Ten cups are fed into a chamber simultaneously. Steam is admitted to this chamber, and the cups are released after 4–6 s of exposure. They are transported into a buffer storage chamber, the sterility of which is maintained by an overpressure of sterile air. Then, one by one, the cups are transported to the filling station while the next batch of 10 cups is sterilized.

The lid material is simultaneously passing through a different pressurized steam chamber in which a length of web corresponding to 10 lids is sterilized using the same conditions as described above. The sterilized lid material is fed into a magazine that is kept under overpressure of sterile air and from there continuously to the filling station. After the lid is sealed to the filled cup, the container leaves the sterile area on a conveyor belt. Though the sterilization of cups and lids is a batch process, the filler operates on a continuous basis.

Sterilization by UV Irradiation. The efficiency of UV irradiation sterilization has been questioned, resulting in a recommendation that the application of this sterilization procedure should be restricted to aseptic packaging of

high-acid products (*Verpackungs-Rundschau*, 1981; *Neue Verpackung*, 1980; *Nordeuropeisk Mejeri tidsskrift*, 1979), i.e. liquid food products having pH 4.6 or less (Buchner, 1983; Food and Drug Administration, 1980). On the other hand, it is also believed that UV irradiation sterilization is sufficiently effective to be used for the sterilization of food contact surfaces in aseptic packaging of low-acid food products (Mann, 1980; Möller, 1981).

UV irradiation sources are usually BBC lamps emitting high-intensity UV-C light. Irradiation intensity is usually in the range of 100–250 mW/cm², the radiation source being ca. 40 mm from the irradiation object. In order to cover the entire interior of the cups, up to three lamps are being applied. The time of irradiation of each cup is in the order of seconds.

The effectiveness of UV irradiation is influenced by a number of factors such as dust, cell aggregates, humidity, etc.

Chemical Sterilization. In some filling systems using prefabricated, plastic cups hydrogen peroxide is used for sterilization of the food contact surfaces (*Deutsche Milchwirtschaft*, 1974a; Mann, 1975b). A 35% solution of hydrogen peroxide with or without a wetting agent is sprayed into the cups, which are subsequently dried by hot sterile air at a temperature of 100 °C (*Deutsche Milchwirtschaft*, 1974a) or 150–200 °C (Mann, 1975b), respectively. The lid material is either sterilized by heat (infrared heating) alone (*Deutsche Milchwirtschaft*, 1974a) or by a combination of hydrogen peroxide and hot sterile air (Mann, 1975b).

Presterilized Cups. Portion cups (10–15 mL) made of polystyrene are presterilized by ethylene oxide. After being transferred to the filling machine under aseptic conditions, the cups are filled and sealed in a sterile surrounding that is maintained by an overpressure of sterile air (*Deutsche Molkerei-Zeitung*, 1971).

3. Aseptic Form-Fill-Seal Cups. Form-fill-seal fillers operate from roll-stock material for both the body and the lid of the cups. Lid and body stock are sterilized either by heat alone or by a combination of hydrogen peroxide and heat. The chamber in which the filling operation is carried out is usually sterilized by a hydrogen peroxide spray followed by drying with hot sterile air. Sterility of the cabinet during production is maintained by an overpressure of sterile air. Air sterilization is usually done by incineration or filtration (HEPA). Piping, valves, etc., are sterilized by saturated steam prior to the start-up of the filling operation. The lower part of the sterile tunnel, through which the packaging material is fed into the filling area, as well as the actual sterile chamber, where filling is taking place, may either be provided by the web of container bodies (Toledo and Chapman, 1973) or be manufactured of stainless steel (*Verpackungs-Rundschau*, 1983; *Deutsche Milchwirtschaft*, 1973, 1974a; Buchner, 1976, 1978a, 1980, 1983; Cerny, 1978; Hansen, 1975b; Linke, 1971; Lütkemeyer, 1983; Mann, 1975b, 1978, 1980), the cups being transported by a conveyor belt through the filler.

Sterilization of the Packaging Material by Heat. In the process of transforming the flat, plastic packaging material web into cups, a thermoforming procedure is used to soften the plastic material prior to forming. The temperatures applied are in the range of 140–200 °C, which is regarded to be the "prime agent for microbiological kill" (Kelsey, 1974a,b). However, this is dry heat; it can hardly be considered sufficient for packaging of low-acid food products.

Saturated steam at a temperature of 165 °C and a

pressure of 6 bars is used for the sterilization of the food contact surface (polystyrene and aluminum cover foil) in addition to the thermoforming temperature of 130–150 °C (Cerny, 1982). By cooling the outside of the forming device, deformation of the cups could be avoided during the 1.4–1.8 s of exposure time necessary.

Chemical Sterilization. So far, hydrogen peroxide seems to be the only chemical sterilant used in form-fill-seal aseptic cup fillers.

Both body and lid stock being supplied in reels pass through a bath of hydrogen peroxide that operates at ambient temperature. The concentration of the hydrogen peroxide is usually 30–35%. A wetting agent is not needed (Buchner, 1976, 1980; Hansen, 1975b). Pressure rollers effect accurate dispersion of the hydrogen peroxide solution over the entire web surface (O'Connell, 1974). In some systems, high-velocity ("jet stream") washing of both the body and lid foil is applied by rapid circulation of the hydrogen peroxide solution. By this procedure air bubbles are removed from the surface and also the microbial load is reduced (*Verpackungs-Rundschau*, 1976a, 1981; Buchner, 1976, 1980). Removal of the hydrogen peroxide from the food contact surface is done by heat applied in the thermoform station (O'Connell, 1974; Toledo and Chapman, 1973) as well as by a flow of hot sterile air (*Deutsche Milchwirtschaft*, 1974a; Linke, 1971). The temperature, thus reached, also contributes to the sterilization efficiency of the hydrogen peroxide treatment. Sterile air blown with a velocity of 0.45 m/s is used to remove the vapors of plastic materials and hydrogen peroxide that unavoidably originate in the deep-drawn operation (*Deutsche Milchwirtschaft*, 1973). Aseptic conditions are maintained in the filling area by sterilizing the plastic web material on its way into the in-feed section, sterilizing the lid material on its way to the sealer, and keeping the interior of the machine free from germs by a flow of pressurized sterile air (O'Connell, 1974).

4. Aseptic Plastic Bottles. Plastic bottles are produced from granulate, usually low- or high-density polyethylene, though other suitable plastic materials can also be used. It is assumed that the pressure (ca. 400 atm) and the temperature of extrusion (ca. 230–240 °C) are sufficient to sterilize the material (Dairy Industries, 1972; *Neue Verpackung*, 1971; Ashton, 1972; Mann, 1975b; Zimmermann, 1975). Though this has been slightly questioned (Lubieniecki-von Schelhorn, 1970), it may suffice for practical purposes (Voss, 1974).

The plastic granulate is extruded into a tube that subsequently is blown to bottles by sterile air. Sterilization of the air is done by filtration (*Deutsche Milchwirtschaft*, 1982; Dairy Industries, 1972; *Neue Verpackung*, 1971; Ashton, 1972; Mann, 1975b; Zimmermann, 1975).

At present, two different procedures are applied in producing aseptic plastic bottles: production of the bottles and aseptic filling in one operation; production of the bottles and aseptic filling in two separate operations.

Production of Bottles and Aseptic Filling in One Operation. In this form-fill-seal operation only the filling device needs to be sterilized prior to start of the filling operation since the sterile surrounding is produced and maintained by the extruded plastic tube itself. Sterilization of the parts of the filling pipe coming into contact with the sterile product is done by steam at a temperature of about 140 °C (Zimmermann, 1974). The product filling pipe extends through the blow pipe that in turn is inside the extrusion head. Product is dosed by a piston filler. After blowing and filling the bottles, the blowing-filling unit is raised and the bottles are closed by a special closing

device, the plastic still being hot enough to permit heat sealing (*Deutsche Milchwirtschaft*, 1974b; Dairy Industries, 1972; *Neue Verpackung*, 1971; Ashton, 1972).

Production of Bottles and Filling in Two Separate Operations. Separating blowing of the bottles and the actual aseptic filling is regarded to simplify the operation from a technical point of view and makes it easier to adapt the two processes to each other (*Deutsche Milchwirtschaft*, 1982). Such a procedure, however, requires a separate aseptic filling unit.

Plastic bottles are blown and closed, the assumption being made that the inside food contact surface is sterilized by the extrusion process (*Verpackungs-Rundschau*, 1976b). Blowing of the bottles is done by using sterile air. The bottles are then either stored or directly transported to a separate aseptic filling station that executes an open-fill-close operation.

Presterilization of the filling station, piping, valves, etc., is done by using live steam and by spraying a suitable disinfectant into the filling chamber followed by drying with hot sterile air. Sterility in the area is maintained by an overpressure of a laminar sterile air flow (*Verpackungs-Rundschau*, 1976b).

The bottles enter the sterile filling chamber through a bath containing a suitable disinfectant in order to sterilize the outside of the containers. The closure is cut-off; the bottle is filled and reclosed by a suitable laminate usually containing aluminum foil as lid material. Sterilization of the closure foil is done either by passage through a bath containing hydrogen peroxide and subsequent heating or by UV irradiation (*Verpackungs-Rundschau*, 1976b).

5. Aseptic Packaging in Prefabricated, Paper-Based Laminated Cartons. In aseptic filling systems operating from prefabricated blanks, the side seam of the carton is already done by the supplier of the packaging material. The blanks are fed into the filling machine, erected, and sealed at the bottom of the carton. Such filling machines are usually adjustable within a certain range of filling volume (*Deutsche Milchwirtschaft*, 1983). The capacity of the filler is determined by the number of parallel filling lines—ranging from 1 to 4—and varies between about 2500 and about 10000 cartons/h.

The erected, bottom-sealed cartons are fed into a sterile chamber. Sterilization of this area is done by spraying hydrogen peroxide onto all surfaces and subsequent drying with hot sterile air. Air sterilization is usually done by filtration (*Deutsche Molkerei-Zeitung*, 1975; Hedrick, 1973; Lisiecki, 1971). During production, sterility of this area is maintained by an overpressure of cold sterile air. The erected cartons are sprayed with hydrogen peroxide, a 30–35% solution being used (Douglas, 1981; Hedrick, 1973). Special “nebulizers” have been developed in order to cover the food contact surface totally (*Food Engineering*, 1981). When the coverage of the inner carton surface was checked, only 30–40% was found to be covered by small hydrogen peroxide droplets. In spite of this observation, good killing efficiency is reported on the entire surface due to the action of hydrogen peroxide in the gas phase (Ashton, 1972; Cerny, 1978).

The amount of hydrogen peroxide applied depends on the size of carton to be sterilized and varies between 0.1 and about 0.25 mL/container (Douglas, 1981; Hedrick, 1973; Lisiecki, 1971). The size of the hydrogen peroxide droplets on the inner carton surface is stated to be about $(3-6) \times 10^{-2} \text{ mm}^3$ (Ashton, 1972; Cerny, 1978). After about 3 s of exposure time, the hydrogen peroxide is dried by sterile air of a temperature of 180 or 205 °C (Cerny, 1978; Douglas, 1981; Gruber and Ziemba, 1970; Hahn, 1981;

Hedrick, 1973; Lisiecki, 1971). The drying period is extended over 7 s (Douglas, 1981) or over a period of 9.3 (Gruber and Ziemba, 1970; Hahn, 1981; Hedrick, 1973; Lisiecki, 1971). An inside surface temperature of about 85 °C is reached in the process (Cerny, 1978). Thus, sterilization of the food contact surface is terminated, and the hydrogen peroxide applied is removed from the treated surface.

The sterilized cartons are moved to the filling station where product is dosed into the containers. Foam, resulting from the filling operation, is removed by a defoaming device using suction procedures (Douglas, 1981).

Finally, the top seam is affected either by hot air sealing (*Verpackungs-Rundschau*, 1982) or by ultrasonic sealing procedures (*Deutsche Milchwirtschaft*, 1975a, 1983). The sealed cartons leave the sterile chamber, and the process of aseptic packaging is terminated.

6. Aseptic Packaging from Roll-Stock Paperboard-Based Laminates. Aseptic packaging systems using paperboard-based laminates from roll-stock are form-fill-seal machines using hydrogen peroxide as a sterilizing agent. Heat is applied in order to increase the sterilizing efficiency of the hydrogen peroxide as well as to eliminate the agent from the food contact surface.

If necessary, a stainless-steel superstructure protects the sterilized packaging material from reinfection. Prior to production, this housing is usually sterilized by a spray of hydrogen peroxide and subsequent drying by hot sterile air. During production, sterility is maintained by an overpressure of sterile air.

In order to sterilize the packaging material contact surface, two different procedures of hydrogen peroxide application are being used: application on the food contact surface only; passage of the material through a dip-in bath.

Application on the Food Surface Only. The concentration of hydrogen peroxide used in such systems varies between 15 and 35%. A wetting agent is needed if a film of hydrogen peroxide is applied onto the hydrophobic polyethylene food contact surface. Depending on food legislation, different wetting agents may be used such as polyoxyethylene-sorbitane-monolaurate (PSM, Tween 20), sugar esters, etc. The sterilizing solution is put on the packaging material surface by a system of transfer rollers. By pressure roller excessive hydrogen peroxide is removed, leaving a very thin layer on the food contact surface, its thickness being on the order of magnitude of microns. By subsequent heating, the sterilizing efficiency of the hydrogen peroxide is increased, and simultaneously, it is evaporated. Heating may be accomplished by passing the material over the surface of a heated drum (*Packaging Technology*, 1983; Mann, 1977, 1978), by radiation heating (B. von Bockelmann, 1978a; Buchner, 1978a), by air (Cerny, 1978; Kelsey, 1974a,b), or by hot air (*Deutsche Milchwirtschaft*, 1975a; *Deutsche Molkerei-Zeitung*, 1975; Brody, 1971; Hansen, 1975a; Mann, 1975a; Schulte, 1981). In these systems, temperatures of about 110 °C are reached on the food contact surface.

Application by a Dip-In Bath. In order to sterilize the packaging material food contact surface by liquid hydrogen peroxide in a bath, elevated temperatures are needed. In such systems usually temperatures of 60–80 °C are applied with exposure times of 6–8 s (*Deutsche Milchwirtschaft*, 1975a; *Deutsche Molkerei-Zeitung*, 1975; Ashton, 1972; B. von Bockelmann, 1970; Brody, 1971; Buchner, 1978a,b; Hansen, 1975a; Mann, 1969, 1978; Samuelsson and Kristiansen, 1978; Schulte, 1981). Excessive hydrogen peroxide is usually removed by pressure roller in combination with air knives, fed with hot sterile air, and/or by hot sterile

air alone. The form-fill-seal operation is done intermittently or continuously, transversal sealing being usually effectuated below the level of the filling product.

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Estimating Thermal Degradation in Processing of Foods

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Breakdown of heat-labile constituents in foods is approximated as a first-order chemical reaction, mathematically similar to the destruction of bacteria. Experimental time/temperature histories of several processes were each transformed into a single degradation value with known or assumed temperature-response (z) values. With this simple procedure, processes and processing steps that were major contributors to thermal degradation of desirable attributes could be identified and modified to minimize loss of quality. Examples are given to illustrate the procedure and some of its applications.

INTRODUCTION

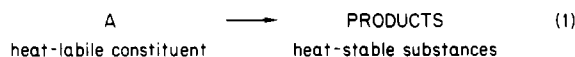
Thermal processing is one method by which fresh foods, limited in both time and space, are preserved and made available out of season or remote from growing areas. The purposes of processing are to destroy pathogenic and/or spoilage-causing microorganisms, to inactivate natural heat-labile toxins and enzyme systems that cause degradation in the food, and to achieve a desirable texture. However, while thermal destruction of the detrimental elements is occurring, nutrients and other desirable attributes are being simultaneously destroyed at varying rates (Ball and Olson, 1957; Stumbo, 1973). The rate of destruction depends primarily upon the susceptibility of the microorganisms, enzymes, or nutrients to degradation by heat. In general, the susceptibility of microorganisms to thermal destruction is much greater than that of enzymes or nutrients. This difference in susceptibility, combined with the heating characteristics of foods, has led to the development of a variety of thermal processing methods, all of which aim at optimizing the preservation of foods.

This paper is not a basic study of the reactions that take place when food is exposed to heat. Rather, it is a practical look at the exposure of food to heat during processing, to identify the types of degradation and the places where they occur. It is an attempt to quantify degradation by a method that can be applied for optimizing processes for a particular food product or can serve as a tool in evaluating the application of heat and the resulting thermal degra-

degradation in existing processing systems. Admittedly, the procedure is crude for the sake of simplicity and general usability, but it points out differences between processing methods and highlights the steps in a process where undesired thermal degradation and waste of energy occur.

THEORY

Consider a heat-labile constituent. It may be a vitamin, or color, or an enzyme, etc. Upon heating, this constituent changes or breaks down into products that are stable under the given conditions.



The first approximation is to treat this breakdown as a first-order chemical reaction (Alberty and Daniels, 1979), and the rate of disappearance of the labile constituent can be expressed as

$$-dC_A/dt = kC_A \quad (2)$$

$-dC_A/dt$ is the rate, the change in concentration of the constituent (dC_A) in a time interval (dt), and k is the reaction rate constant.

Equation 2 may also be expressed by using the D value concept, where D' is analogous to the D value commonly used in thermal process calculations to characterize microbial destruction rates:

$$d \log C_A/dt = -1/D' \quad (3)$$

$D' = 2.3/k$ is analogous to the decimal (90%) reduction value in the thermal destruction of bacteria. D' is a function of temperature, approximated empirically by

$$D'_T = D'_{T_{ref}} 10^{(T_{ref}-T)/z'} \quad (4)$$

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