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RESIDUAL GAS EFFECTS ON HEAT TRANSFER IN OVERPRESSURE
PROCESSING OF FLEXIBLE PACKAGES

by

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B.Sc., University of Alberta, 1982

A THESIS SUBMITTED IN PARTIAL FULFILLMENT OF THE
REQUIREMENTS FOR THE DEGREE OF
MASTER OF SCIENCE

in

THE FACULTY OF GRADUATE STUDIES
(Department of Food Science)

We accept this paper as conforming
to the required standard

THE UNIVERSITY OF BRITISH COLUMBIA

April, 1986

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ABSTRACT

Experiments have been performed to test effects of entrapped air on heating rate indices (f_h) of test pouches subjected to variable conditions in overpressure retort processes. Teflon bricks with centrally located thermocouples were employed as a test material. Water was added within the pouch to provide vapor pressure which would simulate the water activity of a food. Air was injected by means of a hypodermic syringe through a silicone rubber droplet on the surface of the pouch which formed a septum. With repeated injections, air volumes were varied from 0 to 35 mL. Processing temperatures of 115, 120 and 125°C were studied under pressure levels corresponding to 65, 75 and 85% steam in a steam/air mixture. Three retorts were employed: (1) an industrial-scale horizontal Lagarde steam/air retort, (2) a pilot-scale vertical positive flow steam/air retort and (3) the same pilot-scale retort using superheated water with air overpressure.

The heating rates of teflon bricks without retort pouch packaging were also studied at the different temperatures and pressures; analysis of variance revealed no significant differences ($p>0.05$) among conditions of temperature and overpressure level. There were, however, significant differences among the retort systems used. In general, bricks tested in the Lagarde forced circulation steam/air retort demonstrated more rapid heating than in the pilot-scale retort with both media types.

Relationships of heating rate indices as functions of included air volume consisted of either one or two essentially linear segments. One segment formed a plateau region in which there was no change in f_h with

increasing volumes of included air. The second section, apparent with some conditions only, exhibited an increase in f_h as a function of increasing air volumes. A moving regression frame consisting of ten consecutive points was compared against a baseline by covariance analysis. Critical volumes of air were considered to be those volumes above which the f_h vs. air volume function was significantly different ($p < 0.05$) from the baseline. Covariance analysis was performed to compare slopes of the heating rate vs. air volume functions with appreciable data in the second section.

Results of the experiments imply that with an increase in overpressure, larger amounts of air may be entrapped in the pouch with heat transfer remaining unchanged. Air overpressure above 80 kPa allowed up to 35 mL of air to be included with no detrimental effect on heating rates. Increasing overpressure and temperature was shown to display decreasing slopes in the second linear section. Critical air volumes for water/air overpressure processes were substantially higher than those with steam/air mixtures. Steeper slopes were evident on the increasing section with steam/air media indicating a more severe detrimental effect of entrapped air on heat transfer. Studies with retort types demonstrated that processing in the Lagarde retort allowed for larger amounts of air to be included without affecting heat transfer. Predictions of volumes of non-condensable gases which would prevail at retort conditions were made by determining expansion factors from ideal gas relationships. Comparisons of slopes of the $\log f_h$ vs. adjusted air function showed significant differences. Water/air processes in the positive flow retort had the least slope, the Lagarde and positive flow steam/air processes had increasing slopes, respectively.

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NOMENCLATURE

a	half-length of teflon brick (cm)
A	third non-destructive air reading, after processing (g)
a_w	water activity
b	half-width of teflon brick (cm)
B	buoyant force (N); second non-destructive air reading, before processing (g)
c	half-thickness of teflon brick (cm)
C_p	specific heat capacity (cal/g $^{\circ}\text{C}$)
EF	expansion factor
f_h	heating rate index (min)
Fo	process lethality (min)
g	gravitational force (N/kg); difference in degrees between the retort temperature and product temperature ($^{\circ}\text{C}$)
H_{mL}	height of water of each mL in the buret (cm)
H_w	height of water volume (cm)
I	first non-destructive air reading, initial reading (g)
j	lag factor
k	thermal conductivity (cal/s cm $^{\circ}\text{C}$)
Mg	weight of object (mg)
P_1	atmospheric pressure (kPa)
P_2	partial pressure of water at temperature T_2 plus partial pressure of enclosed air (kPa)
P_a	partial pressure of air enclosed (kPa)
P_N	pressure when package is in a state of neutral buoyancy (kPa)
P_R	pressure of retort during processing (kPa)
STP	standard conditions of temperature and pressure

T_1	initial temperature (K)
T_2	temperature of process (K)
V_1	volume of air in pouch at atmospheric conditions (mL)
V_2	volume of air in pouch at processing conditions (mL)
V_m	measured volume of air (mL)
V_{max}	maximum volume to fill the pouch (mL)
V_o	volume of object at STP (mL)
V_{pch}	volume occupied by empty pouch (mL)
V_s	volume of sample (brick and pouch) excluding air (mL)
V_{sa}	volume of sample (brick and pouch) including air (mL)
V_t	volume of object at retort conditions (mL)
W_h	pressure of water level in graduate (kPa)
W_{pch}	weight of an empty pouch (g)
W_s	weight of sample (g)
W_{ws}	weight of package suspended in water (g)
W_{ws+a}	weight of package and added air suspended in water (g)
α	thermal diffusivity (m^2/s); linear thermal expansion factor
β	volume expansion
Δm	difference in mass (g)
Δt	difference in temperature ($^{\circ}C$)
ρ_o	density of object (g/mL)
ρ_{pr}	density of product (g/mL)
ρ_w	density of water (g/mL)

ACKNOWLEDGEMENTS

The author is sincerely grateful to Dr. Marvin A. Tung for his knowledgeable advice and encouragement during the course of this research project and review of the thesis.

She also wishes to thank the members of the research committee, Dr. W.D. Powrie, Dr. J. Vanderstoep, both of the Department of Food Science and Dr. K. V. Lo of the Department of Bio-Resource Engineering for their contribution to the project and constructive review of the thesis.

Much appreciation is extended to H.S. Ramaswamy and Trudi Smith for their valuable expertise and assistance.

The author is grateful to her parents, Jerome and Ann Weintraub for their support and understanding. A special thank you is extended to Benoit Girard for his patience and encouragement.

INTRODUCTION

Innovative food packaging has played an important role in providing consumers with the wide variety of food products that exists today. The research and development of alternate packaging technology is a continual process. One recent development, a shelf-stable thin profile flexible package or retort pouch, was projected to reach \$2 billion in sales by 1980 (Peters, 1975) and earned the 1978 Food Technology Industrial Achievement Award (Mermelstein, 1978). Comparisons of quality and energy requirements place retort pouch products in between traditional canned and frozen foods. Products in pouches may have superior quality as compared to canned goods and processing was reported to be less energy-intensive than freezing (Heintz, 1980).

Since retort pouches have been considered an alternative to canning, comparisons are typically made between pouches and cans. Advantages of the flexible package have been reported by many authors (Tuomy and Young, 1982; Cage and Clark, 1980; Mermelstein, 1976). Shorter processing times required for thin profile packages result in improved taste, color, texture and retention of heat-sensitive vitamins (Davis et al., 1972). Preparation by the boil-in-bag technique is simple and requires little clean-up. The package is easily opened and uses less storage and disposal space. A major concern to food processors is cost. Steffe et al. (1980) reported that the total energy requirement for retorting pouches was 75% less than for cans. A 60% lower energy requirement when processing in flexible packages was reported by Mermelstein (1978). Williams et al. (1983) described reduced material and storage costs associated with retort pouch

packaging. However, initial equipment costs were reported to be high.

Although projections have been made and advantages cited, the use of retort pouches has been limited. European countries and Japan have had reasonable success in their introduction. Production in North America has been limited to small individual companies. Presently, the industry targets for specialty goods or foods for a particular function. Peters (1985) described a line of Smoky Canyon foods produced by Land o' Frost (Lansing, IL) aimed directly toward campers and outdoor enthusiasts. Adams (1984) indicated that there was a bright future in processing of seafood products in retort pouches. Higher priced products such as red snapper and blue crab meat for restaurant use could be competitive with frozen counterparts (Adams et al., 1983). For widespread consumer acceptance of the technology, there must be more exposure to products, perhaps by popular national brands in retort pouches.

Adoption of the technology by large companies has been slow. Kraft introduced an "A la Carte" line which was manufactured in facilities having a relatively low capacity (Anon. 1982), but production was suspended in 1985. Anon. (1982) reported that Nestle and Campbell Soup companies have shown an interest. Hesitation towards large-scale investment may be due to some technological problems such as slow line speeds and the lack of regulations or concrete information regarding aspects of thermal processing. Introduction of materials and technical aspects have made the technology a reality, but additional knowledge of critical factors affecting retort pouch processing is essential.

Beverly (1980) described critical factors as those which affect the

adequacy of the sterilization process, product quality or the economics of the system. Some of the critical parameters are a consequence of the flexible nature of retort pouches. When processing with a flexible film, there is a tendency for the internal pressure within the package to equal the retort pressure. Only a small pressure differential can be maintained. Therefore, as the product temperature rises, the internal pressure may increase and if it exceeds retort pressure to a large degree, the pouch may burst. This is a particular problem during the early stages of the cooling process. Air overpressure processes have been used to overcome this problem. More information is needed on the performance of these media types in relation to their composition.

Another critical factor of utmost importance in thermal processing of pouches is the release of entrapped residual gases from the product during heating. Moreover, there is inevitably a certain amount of air which remains within the pouch when it is sealed. The flexible nature of the pouch allows for expansion of these gases which contributes to the problem of pouch volume increases which may have a detrimental effect on heating behavior. For retort pouch technology to reach its forecasted success, technical aspects must be advanced and critical parameters in thermal processing must be identified and characterized.

Heat penetration studies and process parameters derived may be used to design adequate heat processes. They also provide a means to evaluate heating behavior of products subjected to various critical factors. In a heat penetration test, a time-temperature curve at a given point in the product is recorded. Changes in the amount of thermal energy in a food are produced by transmission of energy from the heating media

surrounding the object (Ball and Olson, 1957). Thermocouples and potentiometric devices are used to sense changes in heat energy. Pflug (1975) and Bee and Park (1978) described many types of equipment available and techniques used to perform heat penetration tests.

Data collected are organized by plotting the temperature difference between the retort and centerpoint (g) on a logarithmic scale as a function of time on an arithmetic scale. Stumbo (1973) discussed different procedures for plotting and evaluating thermal processing parameters. Computer programs have allowed for analysis of heat penetration data in a minimal time (Tung and Garland, 1978). An important process parameter, the heating rate index (f_h) is the time required for the straight line portion of the heat penetration curve to traverse one log cycle. Heating rate indices have been used by many authors (Evans, 1977; Berry and Kohnhorst, 1983; Ramaswamy, 1983) to evaluate critical factors by comparing heating behavior of foods in various package forms.

Because of the need to understand the effects of noncondensable gases within a filled retort pouch on the thermal sterilization process, a study was initiated using a model system. The objectives of this investigation were to:

1. Study the influence of noncondensable gases within a pouch on heating rate indices of materials contained by those pouches,
2. Characterize and compare this influence under different over-pressure processing conditions and retort systems, and
3. Determine limits at which residual gases would interfere with expected heating behavior, as indicated by changes in the heating rate indices.

LITERATURE REVIEW

I Retort Pouch

1. Development of Pouch Material

Development of a pouch capable of withstanding thermal processing began with research into adequate materials and sealing techniques in the late 1950's. Origins of many research projects lie in work done by Reynolds Metals Inc. (Richmond, VA), Continental Flexible Packaging (Chicago, IL) and the U.S. Army Natick R&D Command (Natick, MA). Both Reynolds and Continental manufactured packaging materials and have worked independently or in conjunction with the Natick Laboratories to develop and evaluate suitable materials for the production of shelf stable foods in retortable pouches. The U.S. Army has a vested interest in retort pouches with hopes to completely replace the "meal-combat-individual" or C ration with a new "meal-ready-to-eat" using retort pouches and familiar foods (Mermelstein, 1978).

Early work by Nelson and Steinberg (1956) tested the suitability of polyester (50 and 75 μm) and Trithene (50 and 100 μm) for retorting foods. They concluded that general strength of the films was good but closure seals made with an impulse sealer must be improved. The packages tested were clear laminates, which contributed to eye appeal, but created a problem with gas permeability (Hu et al., 1955; Ayoub et al., 1974).

Keller (1959) reported that laminates containing aluminum showed great promise because of excellent barrier properties as was shown by a polyester/aluminum foil/vinyl laminate. Evaluations of more than 100 packaging materials, including many plastics and combinations, were carried out and it was determined that aluminum foil was an essential

component of the packaging material (Rubinate, 1964). All retort pouches used in North America have aluminum foil as the middle material in the laminate. Foil-free pouches were produced in Japan as reported by Ebben (1979) and by DRG Packaging in England. Products in these transparent packages had a shelf life of six to eight weeks. Typical outer material was polyester, which provided printability and strength. Nylon was suggested but proved to be costly and was sensitive to water (Goldfarb, 1970). The inner layer was commonly polypropylene or a modified polyolefin serving as a heat sealable layer and an inert surface for food contact.

Strength of the flexible material has been tested primarily as a package for military service. Agarwal and Kumta (1974) studied the puncture resistance of flexible packages with different probes. Preliminary studies of rough handling evolved the conventional practice of enclosing the pouch in a paperboard folder. Burke and Schultz (1972) performed rough handling treatment by vibration and drop tests for comparison of pouches and cans. Their results showed no difference between the containers in their capabilities of withstanding rough handling. However, cans resulted in more damage to the outer shipping container.

2. Evacuation Techniques

Removal of noncondensable gases from retort pouches is standard practice. The significance of gas removal will be discussed in more detail in a forthcoming section. Essentially, it is desirable to remove as much gas as possible. Evacuation is performed as part of a multi-function operation; usually in conjunction with filling and/or a

closure-sealing operation (Lampi, 1977). First generation vacuum sealers used preformed pouches. An elementary attempt employed the "snorkel" technique, where a tube with a tapered end was inserted into a short unsealed section of the pouch. Goldfarb (1971) described air removal with this technique. The pouch was purged and a vacuum was applied. When the pouch collapsed, the snorkel was withdrawn. Residual air contents were reported to be less than 5 mL per pouch. This technique presented problems by possible contamination of the seal area with tube withdrawal. Use of mechanical vacuum chambers was more widespread and similar to systems in use for luncheon meats (Lampi and Rubinate, 1973). Two systems commonly used were Swiss-Vac (Hamac-Hansella, Piscataway, NJ) and a design by Koch Multivac (Kansas City, MO). Horizontal conveyors which moved under a hood and closed to form a vacuum chamber, were characteristic of this type. Morris (1981) reported use of a Swiss-Vac system. Pouches were placed manually onto positions resulting in slow production speeds, but a better vacuum was created.

Systems are available implementing steam flush to remove air from the headspace. An improvement in pouch filling speeds from 30 pouches/min for mechanical vacuum to 60 pouches/min with steam flush were reported by Przybyla (1984). Morris (1981) described use of a steam-vacuum Mitsubishi machine by Magic Pantry Foods Inc. (Hamilton, ON). Steam was injected immediately prior to sealing to create a vacuum upon cooling. Fill speeds were faster (rated at 50 pouches per minute) and although poorer air removal occurred, it was well suited to more liquid products. A development by FMC (Santa Clara, CA), the Steam Flow Pouch

Filler and Sealer, was reported by Strasser (1979). This operation involved use of both mechanical vacuum and steam flushing. Pouches were first flushed with steam, then moved to an air-free environment to be filled. An additional jet steaming or addition of inert gas was introduced just prior to sealing. Beverly (1979) demonstrated the air removal efficiency of this equipment. Removal of 40-50% of the original air was found with steam flush alone; furthermore, with the steam tunnel system, 93-95% of air was removed.

Removal of air in other systems was ensured by physical manipulation of the pouch. Tsutsumi (1979b) described a non-vacuum method for packaging viscous liquids. Air within the pouch was reduced to a minimum by stretching both sides of the pouch prior to sealing. Normal operations of opening, filling and sealing occurred. A modification to this type of evacuation involved applying pressure with plates from both sides, creating a squeezing action to force gases out of the package. Narrow bars prevented the product from being pushed up into the seal area. Heid (1970) described a unique system designed with the intent of reducing residual headspace gases. The system, developed and patented in the United States by FMC Corporation (Santa Clara, CA), differed from other processes in that evacuation occurred during processing and the pouch was sealed after cooking. Two clamps held the unsealed portion of the pouch stretched to act as a one-way valve allowing gases to escape. The pouch was submerged in water for heat processing and concomitant exhaustion of residual gases. Sealing was performed after the desired cook. A low level of residual air (1 mL) was reported, however, no industrial application has been reported to date.

A second generation of equipment was developed to form, fill, remove air and seal the pouch. Production speeds of this type of machinery were reported by Cage and Clark (1980) to be approaching 250 pouches per minute which would greatly increase production efficiency. Additional savings could result from the use of roll stock rather than preformed pouches. Lopez (1981) claimed that although equipment costs were higher there would be substantially lower operational costs. At least two systems of this type have been developed. Rexham's Bartelt Div. (Sarasota, FL) introduced a higher speed form/fill/seal machine operating electronically with a microprocessor (Anon., 1980). Another system, horizontal in design, was introduced by Koch Multivac (Kansas City, MO). It was capable of 120 packages per minute, although realistically 90 packages per minute were typical (Anon., 1983). More advances in the technical aspects of retort pouch processing may result in lower production costs, making the technology more favorable.

3. Pouch Seals

Seal integrity has long been considered as a vital criterion for food containers. Thermally processed foods packaged in flexible pouches pose a particular problem due to stresses which develop during heating. A statement by Nelson and Steinberg (1956) that "if the seal area could stand pressure similar to the general surface, a major stumbling block to this type of processing could be removed" illustrated this problem. With the development of new materials and modes of sealing, advances have occurred. A recent study by Roop et al. (1983) reported that failure of pouches during testing was due to the material strength

limitations rather than the seal formed.

Closure seals for preformed pouches and all peripheral seals from roll stock were formed on the previously described filler/sealer or form/fill/seal equipment. A review of heat sealing methods by Brown and Keegan (1973) described techniques for many package types. Typically, for retort pouches, either the hot jaw or impulse seal was used. A hot jaw sealer consists of a bar or plate with an assembled heater on top and a silicone-rubber resilient jaw fixed underneath. A pressure source forces down the upper jaw to create the seal. An impulse sealer utilizes a tension wire supported at each end. Voltage applied to the clamps created a current flow through the high resistance wire which caused heating to form the seal. The contact surfaces were covered with a teflon coated fibreglass cloth. Lampi et al. (1976) summarized criteria for a high performance seal. Fusion of the inner layers was considered to be essential. Assurance that a weld was formed could be tested by tensioning the seal to the point of failure. Signs of delamination would indicate that the film surfaces were fusion sealed. Specifications of tensile strength required by the U.S.D.A. were reported by Shenkenberg (1975) to be 7 pounds per linear inch (12.3 N/cm). Pressurization-hold testing required the ability to withstand 138 kPa (gauge) for 30 seconds. Rubinate (1964) cautioned processors that seal strength was reduced at high temperature and recommended restraint during processing to prevent seal rupture. In addition, there must be no signs of visible aberrations. Military standards required a clean defect-free band across the closure, with a minimum width of 1/16 in (1.6 mm) for an impulse seal and hot bar seal of 1/8 in (3.2 mm)

according to Milleville (1981). Visible defects were thought to result mainly from contamination in the seal area or wrinkling of the pouch material. A special design illustrated by Lampi et al. (1976) used guards which were lowered into the package opening during filling to protect the upper areas. Improved filler nozzle design and high precision filling pumps have helped reduce contamination (Tsutsumi, 1972). Reducing the occurrence of wrinkling has posed another challenge. Stretching of the closure area prior to sealing has helped to minimize fold-over wrinkles.

Despite these improvements, defects occur; however, methods exist to detect them. Visual examination is a common practice. Use of 100% inspection was recommended rather than sampling a random proportion of the pouches (Milleville and Badenhop, 1980). Tsutsumi (1974) reported 0.02% defects from a filling operation with complete visual inspection. Lampi (1977) stated that as a rule of thumb, visual inspections under ideal conditions are 75% effective. A system whereby criteria for rejection were set, based on the severity of defects, was described by Lampi et al. (1976). Small wrinkles or blisters would be considered acceptable, however, occluded particles or large wrinkles must be rejected.

There is a need for development of non-destructive test equipment for on-line inspection of sealed pouches. Infra-red scanning has been described as an effective defect detector (Goldfarb, 1971; Lampi, 1977). The instrument works on transient-heat detection where a contaminated seal will show surface temperature variations by impedance of the heat flux. Details of the design were reported by Lampi (1977). A caliper

measurement was developed which detected irregularities in seal thickness due to contaminants. Gagliardi et al. (1984) designed a computer-aided video inspection system for quality control of pouch seals. An acquisition module stored an image of the product, then a computer analyzed the image to isolate defects and determine whether they would merit rejection. Mechanical implementation of the decision rejected or accepted the package on-line. This system required extensive programming on criteria for defects and threshold levels of rejection.

Stringent inspection and assurance that proper seals are formed free of contamination or defects, aids processing of retort pouches. If defects occur, weak spots may form in the pouch material resulting in failure from stresses imposed by thermal processing.

II Critical Factors Affecting Processing

As the technology for retort pouches continues to develop, there is an increasing need for establishing processing and packaging specifications. Considerable work has been done setting standards for evaluation of pouch seals and material integrity. A similar effort towards identifying and controlling critical factors during thermal processing has not been apparent (Badenhop and Milleville, 1980). Identification of critical factors is an ongoing process. For example, just recently Smith et al. (1985) reported the role of removal of condensate from vertical retorts as a critical parameter in the well established tradition of processing canned foods in vertical steam retorts. In order to set regulations governing the processing of low-acids foods in retort pouches, studies evaluating critical parameters are crucial.

Factors are generally considered to be critical if they affect the adequacy of the sterilization process, product quality or the economics of the system. Many factors must be considered and, due to the flexible nature of the pouch, additional parameters exist that are not present for rigid containers. Berry (1979) identified a number of critical factors for retort pouch processing. Those factors pertaining to the product are: heating characteristics, pouch size, fill weight and residual gas entrapment. Factors contributed by retort design are racking configurations, the heating media employed, mode of circulation, come-up time and the effectiveness of the cool cycle. Many of these factors will be dealt with in more detail.

1. Residual Gases

Although many critical factors in pouch processing are similar to those in metal can processing, the effects of entrapped gases differ considerably. In thermal processing of rigid cans only a small change in total volume occurs. However, due to the flexible nature of the pouch, a significant change in total volume may result from thermal expansion of the solid, liquids and, particularly, entrapped gases. Lampi (1977) cited reasons for removal of gases as primarily providing preclusion of pouch bursting during retorting, assurance of uniform and predictable heat transfer and product stability. Additional advantages of removal of air are easier detection of spoilage and easier cartoning or casing.

a. Pressure-Volume Studies

One of the first studies regarding air entrapped in flexible packages was performed by Wallenberg and Jarnhall (1957). Their results

showed that a maximum ratio between enclosed air and surface area of the package must not be exceeded in order to prevent bursting. The maximum level differed depending on the conditions of sterilization. When subjected to dry air there was no apparent bursting with a ratio of volume to surface of 2:1 (corresponding to 75% of maximum volume of the pouch) compared to a ratio of 3:1 (or 40% of maximum volume) resulting in bursting when autoclaved with steam. There was, however, the suggestion that microscopic leaks occurred in the dry air trials, relieving internal pressures without bursting. It was also evident from their studies that the resistance to bursting was dependent on the moisture content of the air enclosed in the package. Relative humidities of 100% created high pressures in the order of 80.3 kPa with a sealed package of constant volume. Rubinate (1964) studied the feasibility of thermal processing using flexible packages in 100% steam and a water cook with air overpressure as used for glass containers. Results indicated that for use of pure steam as a heat transfer medium it was essential to remove as much air as possible. A maximum of 10 mL of residual gas in a 155 g pouch permitted processing with a pure steam environment without bursting the pouch. Similar results were found by Keller (1959), but although use of pure steam was possible if enough residual gas was removed, a water cook with superimposed air pressure was preferred.

The flexibility of the films and resulting expansion from entrapped gases during heating prompted studies by Davis et al. (1960) on pressure/volume relations which exist in flexible containers. A special apparatus was designed to measure the expansion in volume of packages

containing water and air with increasing internal pressure. Internal pressures were found to develop due to an increase in the vapor pressure of the water, an increase in pressure of air in the headspace and expansion of the food product with increasing temperature. These internal pressures were quantified using the ideal gas law, as follows:

$$\frac{P_1 V_1}{T_1} = \frac{P_2 V_{\max}}{T_2} \quad \dots(1)$$

where $P_1 V_1$ and T_1 are pressure, volume and temperature (absolute) of the air before processing, T_2 is temperature of the heating process and $V_{\max}$, the maximum volume of the pouch. P_2 is the partial pressure of water at temperature T_2 , plus the partial pressure of the enclosed air. In the experiment, the maximum volume of the bag was assumed to be 105 mL. Good agreement was found between the pressure calculated by the gas laws and the actual pressure created inside the pouch with various volumes of entrapped air. A maximum pressure differential of 2.7-23 kPa developed in a pouch cooled with superimposed air pressure, and yet no failures were found. Data from internal pressures that exist and knowledge of strength of the pouch allow the calculation of optimum filling levels of containers.

The use of overriding air pressure to prevent expansion of retort pouches was described by Whitaker (1971). Ideal gas relationships and thermal expansion of solid and liquid components in a food system of peas and brine were used to calculate pressures in the system. When no air was present, 227 g of product would expand an additional 11.3 mL when processed at 126°C with pure steam. A maximum allowable pouch

expansion of 35 mL was predetermined. Therefore, the volume at retort conditions did not exceed the allowable expansion. Consideration of the same system with 5 mL of air was described. If the product was essentially dry, the volume of air would decrease due to increased pressure of steam within the retort. However, when water was contained in the system, Dalton's law of partial pressures must be considered. As water in the product reached retort temperature, the partial pressure of vapor within the pouch was the same as retort pressure. Partial pressure of air in the system can be calculated assuming a maximum pouch volume of 35 mL and taking into account expansion of the contents. Calculated partial pressures of included air were equivalent to the required overriding air pressures for prevention of pouch bursting.

Yamano (1976) predicted pouch expansion in order to determine the pressure needed to prevent pouch bursting. When the pouch was in a state of equilibrium, the retort pressure was assumed to be equal to the internal pouch pressure. As with previously described experiments, the internal pressure was considered to be due to both water vapor and air pressure. From the ideal gas laws, an expansion ratio of the headspace and a total pouch volume were determined. Expansion ratios of headspace gases at various steam/air ratios were determined at different temperatures. A relationship between partial pressure of air within the retort was plotted against processing temperature for different headspace expansion ratios. A maximum allowable expansion ratio of two for a 165 x 200 mm size pouch containing 250 mL of food and a headspace volume of 50 mL was assumed. From this, an expansion ratio of the headspace could be calculated, and consequently a safe overriding

pressure of 20-30 kPa was determined.

b. Effect on Heating Rate

Besides the problem of pouch bursting, residual gases included in pouches are of concern for safety reasons since a predictable heat transfer is required. Nelson and Steinberg (1956) recognized the potential problem of reduced heat transfer due to expansion of gases in flexible pouches. Expansion of gases would result in an insulating effect from poor heat conductivity of entrapped gases. They recommended that superimposed air pressure (at 220 kPa, gauge) held throughout the process would improve heat transfer because entrapped gases would not be allowed to expand. Results, however, were not quantified.

A study performed at Toyo Seikan Kaisha Ltd. (1973), a producer of retortable pouch products in Japan, described the insulating effect of entrapped gases. Experiments were performed on pouches containing 180 g of a curry product processed at 120°C. The heating rate index, f_h , or negative reciprocal slope of the straight line portion of a heating curve and the time required for a selected process lethality were evaluated. Results demonstrated that increasing headspace gas volumes from 0 to 15 mL caused an increase in f_h values from 6 to 7.2 minutes. With an additional 5 mL to a total of 20 mL, an f_h value of 9.4 minutes was reported. Higher f_h values represent slower heat penetration rates. Longer processing times required for adequate sterilization were reported with larger amounts of air entrapped in the package. A similar result was reported by Kopetz et al. (1979) when institutional size pouches were processed in horizontal orientation. Increases in process time up to 35% had been noticed when 150 mL of

residual gas remained within the pouch. Stratification and formation of an insulating layer accounted for the increases seen.

Predictions by Badenhop and Milleville (1980) that the institutional size retort pouch would replace the #10 can has become of interest. Significant cook time and cost advantages were reported. The low profile of the pouch would improve product quality because less severe heating was required. In a 12 x 15 in (30.5 x 38.1 cm) pouch capable of holding 5 lb (2.3 kg), the cold spot is 0.75 in (19 mm) from the pouch surface. In contrast, the cold spot in a number 10 can with the same capacity is 3 in (76 cm) from the can sides and ends. It was also suggested that the institutional market may be easier to deal with since only a few buyers would be involved in choosing the product. However, institutional size pouches present a particular problem with regard to residual gases, prompting research in this area.

Huerta-Espinosa (1981) investigated the effects of entrapped air content on heat penetration characteristics of pears and green beans packed in institutional size retort pouches. Processing experiments were performed with horizontal retort racking orientation using both constraining (firmly sandwiched) and restraining methods. Required thermal processing times to reach an F_{200F} of 3 min for diced pears and an F_0 value of 5 min for green beans were found to increase by approximately 15% for each 100 mL increase in air content for constrained pouches. Increases were larger when pouches were placed in restrained or unconstrained conditions. Similar results were found by Beverly et al. (1980) from studies with institutional size, 30.5 x 43.2 cm (12 x 17 in) retort pouches processed in confined and unconfined

horizontal orientation. Water was contained in the package with air volumes ranging from 0 to 250 mL and the required process times to achieve an F_0 value of 6 min were determined. Results revealed that when pouches were unconfined there was a 20% increase in required process time with 250 mL of air; whereas, confined pouches displayed a 10% increase. The negative impact of entrapped gases affects the productivity and economics of the system. It appeared to be more drastic if pouches were processed while unconfined on supporting racks.

Another study by Berry and Kohnhorst (1983) focused on entrapped gases in institutional size retort pouches. Whole kernel corn and cream of celery soup were processed for 40 and 50 minutes, respectively, in pouches containing up to 250 mL of residual air. Heating rate indices of the cream of celery soup were found to increase from 33.5 min with no air present to a value of 57.3 min when 250 mL of air was included in the pouch. Broken heating curves were displayed in pouches containing whole kernel corn in brine. The f_h remained about the same with increasing air contents but f_2 from the second straight line portion of the heating curve demonstrated an increase of 67% when 250 mL of air was entrapped.

Racking design was also shown to be a contributing factor to the effects of entrapped gases in studies by Evans (1977). Air volumes of 0, 20 and 50 mL were tested in retail size pouches containing a 7% bentonite suspension. Pouches were tested when processed in restrained (unconfined) or constrained positions at air overpressures of 34.5 and 69 kPa. Results showed that both air content and the method of racking were significant. Increased f_h values of 11% were found when 50 mL of

air was included in a constrained pouch. Restrained pouches showed increases of 19% for pouches containing 50 mL of air compared to 0 mL. Air overpressure was found not to be a significant factor in the levels tested. Ramaswamy (1983) investigated the effects of packaging on silicone rubber bricks thermally processed either in horizontal or vertical orientation under different processing conditions. Entrapped air (15-30 mL) in pouches did not appear to influence heating rates in the vertical constrained orientation but f_h values up to 260% higher were found when processing in the horizontal unconstrained orientation at temperatures ranging from 105-120°C and steam/air ratios above 65%.

The insulating behavior and ultimate decrease in heat transfer rates has made it necessary to provide guidelines regarding allowable volumes of air. Predictable heat transfer must be ensured for adequate processing of foods. Although government agencies have not imposed regulations, some good manufacturing practice specifications exist. Tsutsumi (1974) suggested that 10 mL of entrapped air should be generally accepted. This level gave reasonable assurance of predictable heat transfer and prevented pouch bursting. In a report prepared by Campden Food Preservation Research Association (Milleville and Badenhop, 1980), a maximum air content after sealing of 2% of product volume was recommended for use in England. Reviewers from the United States added additional comments, suggesting that this level was too low and that a 10 mL specification in a single serving size pouch was more realistic. The current MRE program and retail processors have adopted a 10 mL allowable residual gas level. It was suggested by both the English authors and American reviewers that measurement of residual gases should

be a routine in process quality control procedure.

c. Methods of Measurement

The most commonly used procedure to evaluate amounts of entrapped gases is a destructive test. Advantages of this technique are that it is simple to execute and equipment used is readily available. Shappee and Werkowski (1972) described in detail the technique and apparatus used. An opening in the package was made when it was totally immersed in water and the air was allowed to escape and be collected into a measuring cylinder, by displacing the water present. Since the water level below the collected gas within the cylinder was usually above that of the surrounding tank, a correction was applied to the gas volume using Boyles law. Although this test was simple and standard, the destructive nature was a disadvantage when multiple samples were to be evaluated. Non-destructive air measurement would be useful in this respect and some procedures have been investigated.

A non-destructive air measurement proposed by Shappee and Werkowski (1972) was derived from Archimedes' principle and Boyles law. Methodology involved weighing a package while suspended in water and then reducing the pressure in a surrounding vacuum chamber until the entrapped gases expanded, rendering the pouch into a state of neutral buoyancy. At this state, the pouch neither floated to the top nor sank to the bottom. The following equation was used to calculate the volume of air in the pouch at atmospheric pressure:

$$V_1 = \frac{P_N(W_{ws})}{P_1 - P_N} \quad \dots (2)$$

where P_1 was the atmospheric pressure at the time of testing, P_N was the pressure when the package was in a state of neutral buoyancy and W_{ws} was the weight in water of the package at atmospheric pressure. Comparisons between destructive and non-destructive air measurements were performed and a linear relationship was found with a coefficient of determination of 0.996. However, actual air volumes were slightly higher than calculated volumes. This was attributed to differences in daily atmospheric pressure and additional pressure due to the water head in the non-destructive method apparatus having a compressive action on the package volume.

Research by Yamaguchi et al. (1972) determined a close correlation between the same non-destructive measurement and traditional destructive methods. However, vaporization of dissolved gases in foods appeared to affect vacuumization operations for accurate determination of pressure at neutral buoyancy. Ghosh and Rizvi (1982) reported results suggesting that non-destructive air measurements were essentially higher than found with destructive methodology. They attributed differences to difficulties in measuring a true internal pressure at the point of neutral buoyancy. Since the pouch was not ideally flexible, the internal pressure may not be equivalent to the environmental pressure. A multiple regression analysis was used to describe the relationship between internal pouch pressure and pressure of the system at neutral buoyancy. Correction factors were determined to obtain an improved estimate of residual air.

Application of non-destructive techniques, based on the principle of neutral buoyancy, is limited to packages which sink in water. Con-

sidering the possibility of processing products such as bakery goods, a technique was developed by Gylys and Rizvi (1983) to non-destructively measure residual gases in products which float in water. The method involved placing a pouch in a container fitted with a buret on top. Water was added to a level in the buret. Then the entire apparatus was placed in a vacuum chamber. Two different vacuum levels were applied and volume differences in water levels were recorded. Boyles law was used to calculate the amount of gas entrapped. Rizvi and Klemaszewski (1985) further investigated this technique and measured actual internal pressures within the pouch. Equations were developed to calculate the amount of air included as corrected to atmospheric pressure. They also determined that changes in volume registered on the buret must be corrected for expansion of dissolved air in the water. Another technique was described by Huerta-Espinosa (1981). Knowledge of product density and volume of the pouch was required. The test was performed by weighing a pouch while suspended in water, then weighing the pouch in air. Archimedes' principle was used to calculate the volume of entrapped gases.

d. Other Effects of Residual Gases

Entrapped gases, in addition to causing deleterious effects on heat transfer, will affect product stability and quality. These factors must be taken into account when deciding upon residual gas levels for quality control. Analysis of headspace gases in a pouch containing hushed tomato gravy was performed by Yamaguchi et al. (1972). Generally, gases in the headspace after processing consisted of 89% N₂, 10% CO₂, 0.6% O₂ and 0.2% H₂. Oxygen in the headspace was of particular

concern because many oxidative deteriorative reactions exist in foods. Tsutsumi (1974) stated that about 10 mL of remaining air was generally accepted with regard to oxidation.

One of the reactions occurring in foods is the peroxidation of lipids leading to oxidative rancidity. Aluminum in the retort pouch material provides a good barrier to intrusion of oxygen from the atmosphere. Therefore, it may be assumed that the total amount of oxygen is finite and the extent of the reaction cannot exceed exhaustion (Karel, 1974). Other food components such as vitamins, pigments and some amino acids are also oxygen sensitive.

Studies by Toyo Seikan Kaisha Ltd. (1973) revealed that vitamin C retention just after sterilization depended on initial air content in the package. Occluded air volumes of 20 mL resulted in 75.7% retention of vitamin C, compared to 83.7, 92.8 and 95.5% retained with 10, 5 and 0 mL of air, respectively. Pereira (1980) investigated vitamin C retention in cherry pie filling thermally processed in retort pouches packaged under high and low vacuum conditions. Although measurement of residual gases was not performed, it may be assumed that high vacuum conditions created an environment with lower air levels. Results indicated that higher internal vacuum prior to sealing resulted in a small but significant increase in vitamin C retention (measurements taken one week after processing). Ascorbic acid contents of French green beans thermally processed in institutional pouches were evaluated by Huerta-Espinosa (1981). Observations of increased ascorbic acid content with increasing amounts of entrapped air were contrary to findings in other studies and to the author's suggestion that residual gases be minimized.

The same author studied other indices of quality sensitive to entrapped air. Color measurements were performed on diced pears and green beans processed with variable air contents. Increased air contents were shown to have a significant effect on color of diced pears; Hunter L and b values were reduced and a dramatically darker overall appearance was detected. Darker colors were explained by a reduction in total phenolics, implying the formation of polymeric compounds responsible for browning. Hunter results for green beans showed increased lightness and decreased greenness. Loss of color pigment in green beans was due to the loss of magnesium ions from the chlorophyll molecule, thereby forming pheophytin which is pale olive in color. Beverly et al. (1980) reported oxidation which occurred in peaches after 8 months of storage. Samples with insufficient air removal showed severe browning.

Studies have been performed comparing nutrient and quality attributes of foods processed in pouches compared to cans. Young (1984) found processing a luncheon-type ham product in retort pouches retained 16 and 26% more thiamine than canned counterparts. Research evaluating color, flavor and texture of peaches thermally processed demonstrated that quality deterioration during storage could be minimized by using retort pouches compared to cans (Potter et al., 1982). In recognizing the advantages of retort pouches, the problem of air inclusion and its detrimental effects must be considered.

2. Processing Media

In the development of retort pouch processes, efforts were made to prevent problems associated with high internal pressures which occur during processing. Of particular concern were latter stages of the cook

cycle and during cooling because at these stages the internal pressure may exceed retort pressure. Typically, air overpressure in a steam environment or overpressure with water may be employed to reduce pressure differentials. Pure steam processes have been employed for heating, however, air overpressure must be introduced during the cool.

Fundamental studies comparing media types were performed by Pflug (1964) and Pflug and Borrero (1967). Studies on efficiencies of the recognized potential media concluded that 100% steam was superior and presented fewer problems in terms of temperature distribution, come-up and heating rate of containers. The use of pure steam was reported by Tsutsumi (1979a) for high temperature-short time (HTST) and ultra high temperature (UHT) systems in Japan, in which temperatures of 135 and 150°C were employed. Since short cook times were associated with these systems, internal temperatures remained substantially lower than the retort temperature. Air overpressure was essential during cooling. Disadvantages of high temperature processes include the requirement of close monitoring of cook times to ensure desired lethality and the possibility of physical changes of some foods after prolonged storage. Refrigeration in the marketplace and future work in combining temperature and water activity were suggested as remedies to the problem.

Tung et al. (1984a) studied heating rates at high temperatures. A mean f_h value 8.3% higher for nylon and 5.4% higher for silicone rubber were found in bricks processed at 130 and 135°C compared to 105-120°C. Differences were attributed to either expansion of the test brick or a reduced thermal diffusivity value at high temperatures. Processing of retort pouches in conventional canning retorts was reported by Roop and

Nelson (1981). Successful processing of confined pouches at 115.6°C in pure steam was achieved.

Pflug (1964) reported that although water/air and steam/air were potentially more troublesome than processing with pure steam, they may be implemented for provision of overpressure conditions. These two types of media are common in Japan, North America and Europe. Studies have been performed comparing media types, yet no conclusive recommendation can be made because each medium type has been shown to have certain advantages and disadvantages. Pflug and Borrero (1967) reported that, with sufficient circulation, steam/air ratios of 75% or greater provided more uniform temperature distribution than an equivalent water cook with 260 kPa total pressure. Advantages of steam/air processes were cited by a retort company chief engineer as providing energy savings and uniform heat distribution (Anon, 1983). Tung and Smith (1980) described water immersion processes as requiring high water use and a great deal of energy. Surface heat transfer was thought to be potentially lower with water cooks than with steam/air since no phase change was involved in heat transfer. In steam/air processes a high surface heat transfer exists when steam condenses on the package and releases the latent heat of vaporization.

A comparison of steam/air and water/air was performed by Yamano (1976). Heating rates of 30 g pouches containing 25% bentonite cake were evaluated. It was apparent that very long come-up times were associated with water processes. When the water retort reached temperature, f_h values of 8.2 min were observed. Samples processed with steam/air exhibited a heating rate index of 10.2 min. The results

indicated that water may be an effective medium, however, come-up times were much faster when employing steam/air mixtures. Although it was determined that use of steam/air was feasible, and perhaps cheaper, many researchers have recommended water/air processes. Beverly (1979) advocated the use of hot water with overriding air pressure because of higher heat transfer coefficients and ease of control. Overall heat transfer coefficients of heating media were reported by both Beverly (1979) and Lopez (1981) based on theoretical calculations by Pflug (1964). Coefficients for steam, water and 75% steam/ air mixtures were found to be 965, 595, and 497 W/m²C respectively. Overall heat transfer coefficients dropped significantly as the percentage of air in the mixture increased.

In terms of process times required with different heating media, Beverly (1980) reported a longer processing time in steam/air than with water for a 280 g product to reach a target F_0 of 6 min. Steam/air mixtures were reported by Wilson (1980) as being difficult to employ with the necessity of temperature and pressure control, along with adequate mixing. In water/air systems, although the retort response was slow, heat transfer rates to the container were generally better. Research by Kopetz et al. (1979) indicated a 15-16% increase in processing time with steam/air compared to water systems. For this reason, they concluded that water/air was more efficient. Tsutsumi (1979b) reported that water processes were commonly used, particularly for pouches containing large amounts of entrapped air, in order to attain rapid and even heat penetration. He suggested avoiding the low steam/air ratios which are necessary to provide adequate overpressure

for preventing pouches from bursting because of the potential interference by air with heat transfer.

3. Processing Conditions

Optimum steam/air ratios have been a focus of interest. There is concern that processing with high air percentages will cause deviations in heat transfer conditions due to the low specific heat capacity of air and the absence of a condensation phase change as with steam. For example, the overall heat transfer coefficient of air is $16.8 \text{ W/m}^2\text{C}$ compared to $965 \text{ W/m}^2\text{C}$ for pure steam. Increasing steam/air ratios from 75 to 95% were reported by Pflug and Borrero (1967) to improve temperature uniformity and reduce the duration of temperature drops when air was introduced after come-up. An investigation by Pflug et al. (1963) revealed that the f_h of pouches heated at 240°F in 90% steam did not differ from f_h values for pouches processed with 75% steam at the same temperature. Results by Yamano and Komatsu (1969) suggested steam/air mixtures over 70% were practical for use. Heating rates and temperature distribution were found to be almost independent of steam/air composition above 70%. These findings were further supported by work of Toyo Seikan Kaisha Ltd. (1973). Their results showed that decreasing steam/air ratios from 100 to approximately 60 or 70% had little effect on heating rates. However, reduction below 60% steam increased values of the heating rate index dramatically.

Heat penetration studies by Tung et al. (1984a) showed that f_h values increased up to 11% by decreasing the steam content from 100 to 50% in a pilot scale positive flow retort. Conversely, in a pilot scale

commercial retort (Lagarde) steam content was found to be non-significant over that range of composition. Properties of steam/air media were characterized by comparing surface heat transfer coefficients in studies by Tung et al. (1984b) using methodology described by Ramaswamy et al. (1983). Surface heat transfer coefficients in both pilot scale retorts were found to increase in an exponential fashion with increasing steam contents. Coefficients in the order of 2,000 and 12,000 W/m²C were found for 50 and 100% steam, respectively. Tung et al. (1984b) pointed out that the surface heat transfer coefficient gave a valuable estimate of media heating potential, but the overall heat transfer and heat rate indices would provide practical information regarding heating of foodstuffs.

Limited studies have examined efficiencies of water/air cooks with variable overpressure conditions. Preliminary investigations by Kopetz et al. (1979) indicated that overriding air pressure in water/air systems helped to maintain heat penetration rates. Their limited data showed a 6-12% reduction in process time when overriding air pressure was increased from 69 to 172 kPa (gauge). Pereira (1980) studied the effects of water with superimposed air pressure on processing time required to reduce peroxidase activity. A 2.85 D process with an experimentally derived z-value were evaluated for cherry pie filling. Pressure was found to be a significant factor. Generally, pouches processed under 103 kPa of superimposed air pressure required less time for sterilization than when processed with no overriding pressure, with some exceptions. Water circulation velocity was found by Peterson and Adams (1983) to be an important factor when employing water/air processes. An

increase in process time of 9.6% was found from high (6.9 L/s) to low (0.6 L/s) flow rates for 10% bentonite suspensions in institution size retort pouches processed at 250°F with 69 kPa overpressure.

Variable temperature of the processing media was studied by Ramaswamy (1983). Temperature was found not to be a significant factor for the heating rate of silicone rubber bricks processed in a positive flow retort with steam/air media. However, nylon bricks showed a significant difference ($p < 0.05$) in the heating rate indices. A 5.5% higher mean f_h was found at 120°C (23.48 min) compared to a 105°C (22.25 min). No significant differences ($p > 0.05$) were found in heating rates at variable temperatures with either silicone rubber or nylon test bricks when processing in a forced circulation Lagarde pilot scale retort.

4. Package Thickness

Retort pouch products derive their rapid heating characteristics because of the thin profile cross section contributing towards overall benefits. One factor affecting pouch thickness is racking constraints. As previously discussed, confined racks will not permit expansion of residual gases and it was possible to use 100% steam for pouch processing (Roop and Nelson, 1981). In confined pouches, the rack spacing establishes a maximum thickness that the package will attain during processing. Improper confinement due to inadequate circulation of process media between layers may be detrimental to the heating rate of the product.

The expansion of pouch contents (particularly residual gases) and fill weights are important factors when utilizing unconfined racking

designs. Expansion of entrapped gases has been discussed previously. Fill weights are critical because of ultimate differences in pouch thickness. Berry and Kornhorst (1983) found decreased heating rates (higher f_h values) with an increase in fill weight when processing cream of celery soup and kernel corn in brine. Effects were more pronounced for the soup, since natural convection currents were enhanced with large fill weights of corn in brine. Beverly et al. (1980) developed a relationship in order to determine f_h values for several pouch thicknesses. For the product tested, f_h values for thicknesses of 0.59, 0.62, and 0.75 inches were calculated to be 8.4, 9.3 and 13.6 minutes, respectively. Heating rate indices can be compared for various thicknesses by multiplying the f_h value by the square of the appropriate ratio of thickness (Ramaswamy, 1983).

EXPERIMENTAL

I Sample Preparation

1. Test Bricks

Experiments utilizing model systems have proved valuable in comparing factors affecting thermal processing of foodstuffs. A wide variety of materials has been implemented, for example, bentonite suspensions (Pflug, 1964; Peterson and Adams, 1983) and bricks of metal or nylon (Ramaswamy, 1983). Test bricks were fabricated from virgin teflon (Cadillac Plastics, Montreal, PQ) for use in experimental phases. The bricks consisted of two teflon slabs. One slab was channelled to allow for placement of a teflon-insulated 24 AWG copper/constantan thermocouple with a fused end placed at the central location. Each pair of slabs was cemented together with a flexible plastic coating (Plastidip, PDI Inc., Northwood, CA). The groove was filled in sufficiently with the material to prevent thermocouple movement and steam or water from entering. Four machine screws at 5.5 cm away from the centerpoint held the brick together. Additional screws were required around outer edges to ensure that the brick remained sandwiched together, since teflon warped slightly with repeated processing. Two brick thicknesses were studied and will be referred to in future discussions as thick or thin. Final dimensions were: thick bricks, 2.1 cm thick, 11.1 cm wide, 15.0 cm long; thin bricks, 1.3 cm thick, 12.1 cm wide and 15.0 cm long.

Using teflon bricks has many advantages. It heats by conduction with a thermal diffusivity similar to many food products. Table 1 lists physical properties as described by Mantell (1958) and a calculated thermal diffusivity which is comparable to literature values as cited by

TABLE 1

Thermophysical properties of teflon.

Properties of teflon (Mantell, 1958)

Specific Gravity (ρ)	2.1 - 2.3 g/cm ³
Specific Volume (s.v.)	475.9 cm ³ /kg
Thermal Conductivity (k)	6 x 10 ⁻⁴ cal/s cm C°
Thermal Expansion (TE)	10 ⁻⁵ /C° (linear)
Specific Heat Capacity (Cp)	0.25 cal/g C°
Water Absorption (WA)	0

Calculation of thermal diffusivity (Loncin and Merson, 1979)

$$\begin{aligned}\alpha &= \frac{k}{\rho C_p} = \frac{6 \times 10^{-4} \text{ cal/s cm C}^\circ}{2.2 \text{ g cm}^{-3} \cdot 0.25 \text{ cal/g C}^\circ} \\ &= 1.09 \times 10^{-7} \text{ m}^2/\text{s}\end{aligned}$$

Tung et al. (1984a). Another advantage is that it may be assumed there are no thermal degradations or permanent modifications of properties at the high temperatures used with retort processing. Importantly, teflon bricks may be fabricated in an identical manner and be used for numerous trials. This provided consistent and repeatable measurements, thereby reducing variations due to sample characteristics.

2. Packaging

Retort pouches (American Can Co., Neenah, WI; 12 μ m Polyester/9 μ m Al foil/76 μ m Polypropylene) of 16.0 x 23.1 cm outside dimensions and 14.4 x 20.5 cm inside (taking seal into consideration) were used. A droplet (approximately 2.5 mL) of silicone sealant (Dow Corning Corp., Midland, MI)) was applied in a lower corner of the pouch and allowed to cure for at least eight hours. This functioned as a septum through which air was injected and with removal of the syringe needle, an airtight seal reformed.

Pouches were fitted with packaging glands (O.F. Ecklund, Cape Coral, FL). Use of extra gaskets sealing the stuffing box were necessary to prevent gases from escaping through the fixture. In order to ensure an airtight seal, tightening was required on the screw fittings until first signs of gasket deformation appeared. Pouches were prepared with about 25 cm of thermocouple wire extending from the pouch and equipped with connectors (Omega Engineering, Inc., Stamford, CT), since occasionally repackaging was required due to leakage of air into the pouch. This allowed easy removal of the brick and facilitated immediate repackaging into previously prepared pouches. A cotton ball was placed inside the pouch, directly below the septum to aid injection.

Each brick was placed in a pouch and connected to an extension thermocouple. Water (10 mL) was added to the pouch to provide vapor pressure simulating a food material. Since there was no absorption of water by teflon, as indicated in Table 1, water was assumed to be totally free and exhibiting a vapor pressure similar to foods with a water activity approaching 1.0.

Samples were vacuum packaged to remove as much air as possible. For some trials, a Multivac (Model AG-5, Algau, W. Germany) equipped with a narrow bar impulse sealer was employed at the maximum vacuum setting. To ensure an adequate seal for retort processing, an additional seal was formed using a Sentinel sealer (Packages Industries Group, Inc., Hyannis, MA) at operating conditions of: airline pressure 375 kPa corresponding to a jaw pressure of 275 kPa and temperature setting of 227°C. Volumes of air remaining after vacuum packaging were less than 5.0 mL. In other trials, a mechanical vacuum Swiss-Vac machine located at Magic Pantry Foods Inc. (Hamilton, ON.) was used. It was a production model for solid fill foods with speeds rated at 30 pouches per minute (Morris, 1981).

Pouch integrity was tested by allowing 3-8 h to elapse with no appearance of air within the pouch. Leakage could be detected visually since the pouch was packaged tightly against the brick. If signs of leakage were noticed, repackaging was necessary until an airtight sample was ensured.

II Air Measurement

1. Non-destructive Measurement of Air

A non-destructive measurement was useful in these experiments to determine air volumes within the pouch prior to processing. Shappee and Werkowski (1972), and Rizvi and Gylys (1983) described a method of non-destructive measurement as discussed earlier. Procedures involved determining the vacuum pressure within a bell jar necessary for the pouch to achieve neutral buoyancy in water. This method was attempted with results showing inconsistencies and lack of reproducibility. That approach was abandoned and a modified technique was employed to monitor addition of air and possible leakage of gases into or out of the pouch. It was performed by weighing a vacuum packaged pouch while suspended in water. Figure 1 illustrates the apparatus used for this purpose. A scale equipped with a hook on the underside for weighing, mounted on a wooden base with a circular hole and placed over a water basin is depicted. There was clearance between the wooden base and water basin. Pouches were suspended from the hook by use of a clip and totally immersed in water. Water level was kept constant and the scale was zeroed with the clip on.

Once each package was weighed, desired volumes of air were carefully injected through the silicone septum. In each set of trials, target air volumes were administered. For 14 bricks of each thickness,

Figure 1. Apparatus for non-destructive measurement of air.

two bricks were left unpackaged and three bricks were injected with volumes of 0, 10, 20 and 30 mL of air. Each brick was identified by a number, and the bricks were randomly assigned to the various test conditions.

After air addition, samples were weighed in water again. Based on Archimedes' principle, the difference in weight was equal to the volume of air added from the equation:

$$W_{ws} - W_{ws+a} = \rho_w V_s g + \rho_w V_l g - \rho_w V_s g \quad \dots(4)$$

$$\Delta m = \rho_w V_l$$

where W_{ws} is weight of the sample in water and W_{ws+a} is weight of the sample and added air in water; ρ_w the density of water; V_s is the volume of the sample; V_l is the volume of air; and g is the gravitational force. Derivation of this equation is shown in Appendix I. Assuming the density of water equal to 1.0 g/mL, for each mL of air added, the mass of the sample and air will be reduced by 1.0 g. With this assumption, addition of air and any leakage may be monitored. This technique was used only as a relative measure of air volumes. The measurements were performed in three stages:

1. after vacuum packaging,
2. after injecting desired volumes of air, just prior to processing, and
3. after processing, just prior to destructive air measurement.

The difference between measurement two and one, represents the amount of air added by injection. Possible air leakage during processing may be calculated by the difference of measurements three and two. Any pouches

with leakage greater than 5 mL were omitted.

2. Destructive Measurement of Air

Conventional air measurement is a procedure whereby the pouch is opened under water and gases are collected in a volumetric vessel. Since pouch integrity is lost and the air is discharged from the pouch, such methods are generally termed "destructive". Shappee and Werkowski (1972) described the method used in this experiment. Figure 2 depicts equipment assembled to measure entrapped air. A funnel was attached to the mouth of a 50 mL buret to increase the area for entry of gases. As illustrated, the buret was inverted and placed in a water basin. Water was filled to the 50 mL measure by suction with an attached rubber tube. A constant column height above the water level was ensured by consistently immersing to a marking on the funnel.

Testing was performed by carefully immersing the pouch preventing any accumulation of air bubbles on outside surfaces, then an opening was cut in a corner of the pouch while directly under the funnel. Entrapped gases were allowed to escape and be collected. Care was taken to prevent gases from escaping beyond the funnel area. Volumes of gas were measured by displacement of water. A correction for pressure differential due to water height was necessary using the relationship:

$$V_1 = \frac{(P_1 - W_h) V_m}{P_1} \quad \dots(5)$$

where V_1 is the volume of air at atmospheric pressure P_1 . The pressure due to the water level in the buret (W_h) was calculated from:

Figure 2. Apparatus for destructive measurement of air.

$$W_h = H_w - (V_m \cdot H_{mL}) \quad \dots (6)$$

H_w is the height of water from water level to 50 mL mark; V_m is the measured volume and H_{mL} is the height of water of each mL in the buret.

3. Combining Destructive and Non-destructive Measurements of Air

Destructive measurement of air volume on its own, provided the final amount of air after processing. Non-destructive measurements yielded relative volumes at various stages. Combining information from these tests was valuable. For example, destructive results were related to the third (post-processing) non-destructive measurement. From this standpoint, back calculation was performed, assuming a 1.0 mL/g difference from the non-destructive measurements. Equations describing back calculations are shown in Appendix II. A measurement of the weight in water of the pouch post-processing puts non-destructive and destructive measurements at the same reference point. Initial volumes of air (in the vacuum packaged brick) may be determined by differences in these two values. Similarly, a projected weight of pouch in water with no air present can be found by correcting non-destructive air volume by calculated differences.

Each processing set consisted of three runs. Volumes of air for each run were determined. For the first run, the air volume was taken as that prior to processing, back calculated from the final air volume. Air measurements by the destructive method were used for volumes of air in pouches of the third run. An estimation of volumes of air in pouches of run two were calculated by one half the difference between values from measurements before and after processing.

III Processing Conditions

Overpressure processes are characterized by retort temperature and pressure. Operations employing steam and air mixtures refer to these conditions as the fraction of steam (or percentage steam) at designated temperatures. Fractional steam content is calculated as the ratio of saturated steam pressure (absolute) at retort temperature to total retort pressure from steam and air. From this relationship, desired retort pressures at specific temperatures can be determined. Conventional citation for water and air processes states temperature and amount of overpressure.

In this experiment similar retort conditions for both steam/air and water/air processes were studied. Variables consisted of three temperatures: 115, 120 and 125°C, and three pressure levels corresponding to 65, 75 and 85% steam. Pressure levels for water/air processes are referred to as 1, 2 and 3 for each temperature, corresponding to 65, 75 and 85% steam, respectively. Table 2 lists all retort temperatures and pressures. A full factorial design with duplicate runs of each condition (a total of 18 runs) was performed. It was determined that pouches packaged with bricks could withstand at least three consecutive runs. Consequently, experiments were performed in six sets, each with three runs. All runs were carried out in random order.

IV Retorts

Experiments were carried out on three retort systems. Two utilized steam/air mixtures and the third employed superheated water with air overpressure.

TABLE 2

Summary of processing temperatures and pressures studied.

Temperature, °C	% Steam (Pressure Level ¹)	Retort Pressure, kPa
115	65 (1)	259.9
115	75 (2)	225.2
115	85 (3)	198.8
120	65 (1)	305.2
120	75 (2)	264.5
120	85 (3)	233.4
125	65 (1)	356.8
125	75 (2)	309.2
125	85 (3)	272.8

¹as referred to for water/air processes

1. Lagarde Steam/Air

Phase one of the experiments involved processing in an industrial scale Lagarde retort (J. Lagarde, Montelimar, France) located at Magic Pantry Foods Inc., Hamilton, ON. The processing plant was equipped with 4-car horizontal retorts of 1.1 m diameter and 4.6 m length. The same retort was used for all trials. Steam/air mixtures were employed in this system. A 9.3 kW (12.5 hp) turbo fan located at the rear of the retort forced the heating medium toward the door through two side plenums, returning to the fan after passing through the retort load, thereby producing a horizontal media flow pattern. Media flow rates were characteristic of the retort design and therefore were not variable by an operator.

Desired steam/air ratios were achieved by temperature control through steam addition and total pressure by air introduction through pneumatic action valves. Automatic operation of the system was used, although manual control was possible with a switchboard on the front panel. Automatic control was programmed by use of an aluminum card. Process temperatures and pressures for time profiles and any desired ramping were input onto the card. An electric eye detected the desired levels and controlled inputs of steam and air. In automatic operation there was a short venting period after "steam on" when the turbo fan was not in operation. Following the heating cycle, the fan was turned off during cooling when cold water was added to flood the retort. Circulation of water during cooling was ensured by currents created with pumps.

A filled retort holds four cars of racks containing pouches. In

all cases car number four (closest to the door) was used for sample pouches. Ballast cars were placed in all other positions. Racking orientation of the sample car was horizontal and the pouches were "unconstrained". Although considered unconstrained, clearance between racks was approximately 3.8 cm. Trays were made of convoluted expanded metal to improve circulation, however, no spacer levels were present. Sample racks in the fourth and seventh positions from the top were used and fifteen pouches were placed on each level. Other levels were filled with ballast pouches. Each sample pouch was randomly placed (position determined by random number generation) for each set of three runs.

2. Vertical Positive Flow - Steam/Air

A second phase of experiments was carried out using a pilot scale vertical positive flow retort designed for steam/air processing. Details of construction and operation employing steam/air in this system have been reported elsewhere (Tung et al., 1984a; Young, 1984). A homogeneous steam and air environment was created by continual flow of media through the system. It is capable of flow in upward and downward direction; however, upward flow was used in this experiment. Steam and air were introduced at the bottom through a cross spreader and vented through a ring shaped manifold with holes on the underside. The manifold was connected to a vent line at the top of the retort.

Control of the system was attained by a Taylor fullscope temperature and pressure controller (Taylor Instruments Ltd., Toronto, ON). Previously described set points of desired temperature and pressure conditions were adjusted. Proportional valves, regulated through pneumatic action by the controller, maintained the set points. A prop-

portional valve on the steam line maintained the target temperature, while pressure was controlled by a proportional valve on the vent line (from the manifold).

Air was added at a constant rate to the steam line and the media were mixed before entering the cross spreader. Air flow rates were adjusted through a calibrated Flowrator flowmeter (Fischer and Porter Limited, Downsview, ON) with a head pressure of 414 kPa (gauge). The desired medium flow rate was 40 standard cubic feet per minute (scfm) or $68 \text{ m}^3/\text{h}$ at standard conditions of temperature and pressure. Medium flow at this rate was approximately equivalent to five complete changes of the retort environment per minute (Ramaswamy, 1983). To achieve desired media flow rates, rotameter settings were based on the following relationship:

$$\text{Rotameter Setting} = \frac{\text{medium flow rate (scfm) (100\% steam)}}{\text{calibration factor}} \dots (7)$$

A calibration factor of .394 scfm/unit x 100 was previously determined. Using the equation, settings for conditions were 35.5 for 65% steam, 25.4 for 75% steam and 15.2 for 85% steam.

Samples were pressure cooled in all experiments. Cold water was introduced near the retort bottom while maintaining overpressure with air. Elevated pressure levels were sustained for the first part of the cool cycle until samples reached an internal temperature of approximately 70-80°C. Retort pressure was then slowly reduced to atmospheric pressure.

A rack was constructed to provide horizontal positioning. Clearance above each level was adjusted to 3.2 cm by supports to provide

"unconstrained" positioning of pouches. Racks were made with additional spacer levels between sample layers to ensure circulation around the package. Each layer was hexagonal in shape and held five pouches. Six levels were assembled to hold all sample bricks. The final height of the assembly reached approximately 15 cm below the manifold. All samples were adequately immersed during cooling.

3. Vertical Positive Flow - Water/Air

The pilot scale vertical positive flow retort was also modified to implement water/air processing. This would enable comparison of media within the same vessel. Control of temperature and pressure was similar to that described for steam/air operation, except that the retort contained water heated by steam and agitated by air addition through the steam spreader at the bottom. Although the air flow rates were not necessarily equivalent to five changes of retort environment per minute, they were consistent with flow rates used with steam/air processes. Variations in operation occurred during the "steam on" and "start of cool" operations and will be elucidated.

Pflug and Borrero (1967) described use of water processing with air overpressure in a retort of similar design. Their operation procedure involved filling the retort with water, then applying steam to heat the water. Using this technique, come-up times of 17 minutes were reported. This was considered unacceptable for comparison with other systems; therefore, a method was developed whereby preheated water could be introduced to the retort.

The hot water reservoir on an FMC 500W Universal Sterilizer (F.M.C.

Corporation, Santa Clara, CA) was used to pre-heat processing water to desired temperatures. The retort used a pump to circulate cold water to a steam injection port, then back into the storage vessel. Only the reservoir of this system was employed, so valves entering the processing vessel were closed at all times. A 3.8 cm (outside diameter) flexible pipe was connected from the reservoir to the bottom vent line of the positive flow retort for transfer of preheated water. A few preliminary trials determined that water should be preheated to 10C° higher than that desired for processing.

A driving force for the transfer was created by a pressure differential between vessels. Since the difference in height was minimal, additional air pressure (approximately 70 kPa) was introduced to the reservoir just prior to "steam on" ensuring rapid transfer of the heated water. The water reservoir control valve was set to maintain an elevated pressure during transfer. Differential pressure between vessels was upheld by keeping the pressure controller setpoint on the receiving vessel at zero, so the vent line proportional valve remained open during the transfer.

At "steam on" the water transfer valves were opened and the retort temperature controller was simultaneously set to initiate the input of steam to the processing vessel. Once water reached the desired level and all pouches were immersed (as indicated on a sight glass), transfer valves were closed. Operating pressure was then set by the controller and air was introduced.

Cooling operations were similar to steps performed when employing steam/air media with a few distinct differences. After steam was turned

off, hot water was drained from the retort. Care was taken to ensure that almost all hot water was removed without a decrease in pressure which was maintained by addition of air. Cold water was then introduced through the bottom water line and cooling proceeded as described earlier following steam/air processes.

V Data Acquisition

1. Temperature Profile Measurement

Samples prepared as previously described were randomly placed in rack positions determined by random number tables (Khazanie, 1979). The extension thermocouple wire from the sample was connected to corresponding 24 AWG copper constantan wire fitted through the retort. Environmental temperatures were monitored by six thermocouples (of similar wire) with fused tips. In the Lagarde retort, three locations on each of the two sample trays were monitored. In the vertical positive flow systems, two thermocouples were secured on alternate sample layers. All thermocouples with connections were calibrated against the retort thermometer using a steam environment.

A precalibrated pressure transducer (Model A-5/1148, Sensotec, Columbus, OH) was used to monitor retort pressure during processing. It was fitted to a bleeder port on top of the Lagarde retort or a port on the upper side of the vertical positive flow retort. A constant excitation voltage of 10 V (DC) was provided using a Hewlett-Packard power supply (Model #62148, Hewlett-Packard Co., Rockaway, NJ).

A Doric Digitrend 235 (Doric Scientific, San Diego, CA) was used for logging data for experiments performed at Magic Pantry Foods. A Kaye Ramp II Scanner/Processor (Kaye Instruments Inc., Bedford, MA) was

used for experiments performed in the vertical positive flow systems. Channels from thermocouples were programmed to read in °C and the channel from the pressure transducer was read in millivolts. Channel numbers and thermocouple connections were identical for each subsequent set of runs. Readings were taken at one minute intervals from steam on through a 35 minute period (including come-up time). Come-up times of 6-7 minutes were typical in the Lagarde, whereas 4-6 minutes and 3-5 minutes were characteristic come-up times in the pilot scale steam/air and water/air systems, respectively. Cooling data were not collected.

2. Analysis of Heating Rate Index (f_h)

Data were recorded on both a paper strip from the data logger and a magnetic tape. A Columbia 300D Digital Cartridge Recorder (Columbia Data Products Inc., Columbia, MD) connected to the auxiliary I/O board of the data logger through an RS232 cable was used for recording on the magnetic tape. Data on the tape were transferred to an Apple II Plus microcomputer (Apple Computer Inc., Cupertino, CA) for computations using programs developed for this purpose.

One step involved extraction of environmental temperatures for each run. Overall mean temperature from time of stabilization to end of cook was calculated as retort temperature. Retort pressures were taken from the strip tape printout. An average mV reading from minute 15 to minute 30 was calculated. Retort pressure (P_R in psig) of each run was determined by:

$$P_R = \frac{(mV_{\text{mean}} - 11.395)}{0.834} \quad \dots (8)$$

This relationship was previously determined by calibration techniques.

The variable used as a basis for comparison of heating behavior was the heating rate index (f_h), as described earlier. Temperature profiles over time for each brick were extracted and f_h values were determined using a program on the microcomputer. In this program the temperature difference between retort and center point (g value) was plotted on a logarithmic ordinate as a function of time. Heating rate index was evaluated as the negative reciprocal slope of a straight-line portion of this line determined by the method of least squares. The heating rate index (f_h) represented the time (in minutes) required for a log reduction in g value. Lower f_h values indicated faster heating rates.

Ramaswamy (1983) reported broken heating curves in some samples of packaged bricks; as well, Berry and Kohnhorst (1983) found a similar behavior. Slightly broken heating curves were apparent for some samples, particularly those with large amounts of included air. For these samples, limits chosen to represent the "straight-line" portion were taken from a consistent interval of g value on the ordinate.

3. Data Treatment

For each set of conditions, heating rate indices were measured as a function of included air volumes. Due to variation in the amount of air after vacuum packaging and small but measurable leakage during processing, a continuum of air measurements resulted. Air volumes were not exactly as targeted for each of the duplicate runs. Therefore, to ensure a homogeneous continuum, duplicate runs were combined.

Regression analysis was used to describe the effect of residual

gases on heating rate indices. Comparisons of these relationships between factors studied in the experiment were performed by analysis of covariance (Snedecor, 1965). Analysis of variance was done for all conditions on bare bricks included in each run.

Computations were performed for analysis of variance and covariance analysis by UBC MFAV (Le, 1978). Both program packages were available on the UBC Amdahl 470 V/8 computer.

RESULTS AND DISCUSSION

I Air Measurement

The non-destructive measurement of air utilized provided a fast and reliable method of monitoring residual gas entrapped in a package. The weight of pouches in water were measured to within 0.1 g. Consequently, volume changes to 0.1 mL can be detected. Back calculations as previously described were based on coupling information from non-destructive measurements and volumes quantified by destructive air measurement. The resultant "no air weight" was valuable in comparison with subsequent readings.

Presently, industry performs destructive measurements as a routine quality control test for residual air in pouches. Since the analysis is destructive in nature, only a small representative sample is tested. In addition, opening a pouch immersed in water renders the contents unusable for other quality control purposes. Non-destructive air measurements as discussed by Shappee and Werkowski (1972) could be acceptable. However, as found in this study, some methods can be difficult to employ and results may be unreliable. There is a potential for an "on-line" non-destructive air measurement applicable to automated packaging with specific fill weights. Huerta-Epinosa (1981) utilized such a method for air measurements based on Archimedes' principle. It required a known density of the product to be packaged and a value of weight and volume contributed by an empty pouch. These would be constant values for a homogeneous product. Consequently, from fill weight or weighing the package in air, the following relationship applies:

$$V_s = \frac{W_s - W_{pch}}{\rho_{pr}} + V_{pch} \quad \dots(9)$$

where V_s is volume of the sample excluding air, W_s and W_{pch} are weight of the sample and weight of an empty pouch respectively, V_{pch} is the volume of the pouch and ρ_{pr} is the product density. The package volume excluding air could be determined. If fill weights were constant, this may be a constant value and not require an additional measurement. Next, the package would be weighed while suspended in water. A volume as contributed by the package, contents and air is calculated from:

$$V_{sa} = \frac{W_s - W_{ws}}{\rho_w} \quad \dots (10)$$

where V_{sa} is volume of sample and air, W_{ws} is the weight of the sample when suspended in water and ρ_w is the density of water. Therefore, volume of air entrapped within a pouch can be calculated from the difference between the two values found in equations (9) and (10).

If fill weights were adequately consistent this analysis may be performed by only obtaining the weight of the pouch in water. It could be performed just as the pouch left the vacuum sealer. A conveyor belt would immerse the pouches in a shallow trough and random samples would be suspended and weighed. Other industries have implemented the buoyant effect as a criterion for grading products; for example, peas are placed in a flotation tank containing brine to separate mature from green products (Cruess, 1958).

II Unpackaged Bricks

Unpackaged bricks were included in each run. They provide a means by which factors may be studied independently without the effects of residual gases. An analysis of variance was performed on the factorial experiment for thin and thick bricks separately. Results of the analysis are tabulated in Table 3. No significant difference ($p > 0.05$) was found in heating rates for variables of temperature and steam percentage (pressure level). This is consistent with the findings of Ramaswamy (1983) where similar factors were found non-significant ($p > 0.05$) for nylon bricks in both vertical positive flow and Lagarde retorts. This finding demonstrates that all three pressure levels and temperatures have the same efficiency of heating on an unpackaged sample which excludes the potential effects of the pouch and residual gases.

A significant difference was seen in the data from different retorts. Duncan's multiple range test was performed and results are reported in Table 4. In regard to thin bricks, heating rates in the Lagarde retort were significantly faster (low f_h values) than were evident with either water/air or steam/air in the pilot scale retort. All retorts were found to be significantly different from each other when processing thick bricks. Highest mean f_h values were found in the positive flow retort employing steam/air. Water/air media in the same retort yielded slightly lower heating rate indices, and values found from processing in the Lagarde system were lowest. It is evident from these results that processing of unpackaged bricks in the Lagarde system may provide more efficient heat transfer than water/air or steam/air in vertical positive flow systems.

TABLE 3.

Analysis of variance for heating rate indices
of unpackaged teflon bricks.

Source of Variation	Thin		Thick	
	df	F-Ratio	df	F-ratio
Retorts	2	6.46**	2	20.94**
Temperature	2	1.57 ns	2	0.15 ns
Steam %-Pressure level	2	1.55 ns	2	0.47 ns
Interactions				
Retorts - Temperature	4	1.11 ns	4	0.07 ns
Retorts - Steam	4	0.38 ns	4	0.71 ns
Temperature - Steam	4	0.71 ns	4	1.04 ns
Error	81		81	

ns not significant ($p > 0.05$)

** significant at $p < 0.01$

TABLE 4

Duncan's multiple range test for heating rates of unpackaged bricks in different retorts.

Retort	Thin Bricks		Thick Bricks	
	f_h (min)	Duncan Test ¹	f_h (min)	Duncan Test ¹
	mean (std. dev.)		mean (std. dev.)	
Lagarde	6.08 (0.31)	a	17.78 (0.80)	a
Steam/Air	6.42 (0.51)	b	19.24 (1.09)	c
Water/Air	6.46 (0.65)	b	18.32 (0.83)	b

¹Values with the same letter within a column are not significantly different ($p>0.05$)

Previous studies have found large variability in f_h values. Ramaswamy (1983) found coefficients of variation for heating rate indices of unpackaged bricks ranging from 4.5-5.1% for nylon and 6.4% for silicone rubber. Heating rate indices for pouches containing whole kernel corn in brine with essentially no residual air were reported by Berry and Kohnhorst (1983) with coefficients of variation of 9.9%. As indicated in Table 5, similar coefficients of variation in sets of data from each retort vary from 5-10% for thin bricks and 4.5-5.7% for thick bricks. In contrast, experiments by Patino and Heil (1985) on heat penetration with potatoes in brine (no volumes of air reported) showed coefficients of variation in the order of 15.9-25.5%.

A value of thermal diffusivity (α) may be calculated from measured heating rate indices and brick geometry by the relationship reported by Olson and Jackson (1942):

$$\alpha = \frac{.933}{f_h \left(\frac{1}{a^2} + \frac{1}{b^2} + \frac{1}{c^2} \right)} \quad \dots (11)$$

whereby a, b and c are one-half brick thickness, length and width respectively. The average calculated thermal diffusivities were $8.82 \times 10^{-8} \text{ m}^2/\text{s}$ for thick and $1.02 \times 10^{-7} \text{ m}^2/\text{s}$ for thin bricks compared to $1.09 \times 10^{-7} \text{ m}^2/\text{s}$ calculated from thermophysical properties of teflon in Table 1. Differences may be due to the composition of teflon from which the bricks were made or possible effects of the plastic rubber compound used to secure thermocouples at brick center.

TABLE 5.

Coefficients of variation for heating rate indices
of unpackaged bricks.

Brick Thickness	Retort	Coefficient of Variation, % (std. dev./mean) x 100
Thin	Positive flow steam/air	8.0
	Positive flow water/air	10.0
	Lagarde	5.2
Thick	Positive flow steam/air	5.7
	Positive flow water/air	4.5
	Lagarde	4.5

III Packaged Bricks: Included Air

Heating rate indices (f_h , min) were found for all test packages containing air processed in retort conditions as previously described. Figures 3 and 4 are examples of data collected from thin and thick bricks processed in the Lagarde retort. All other relationships are illustrated in Appendix III (a, b, c and d). By observation of these figures, a general trend in the data was evident. It appears that some conditions, particularly those of 65% steam (pressure level 1) showed no effect of included air volumes on heating rate indices. These relationships form essentially a horizontal line. Data for some other conditions (usually 75% steam, pressure level 2) appeared to consist of two sections; one being a horizontal line similar to the relationship described above and a second part demonstrating an increase in f_h as air volumes increase. This trend was apparent in many conditions. Another type of relationship was observed for conditions of 115°C and 85% steam (pressure level 3) with thick bricks. There is no apparent section of plateau and f_h values increased at very low levels of included air. Therefore, the relationship of f_h as a function of air content can be described as containing either one or both sections of a two part model. One part exists as a plateau demonstrating that air volumes have no effect on f_h ; the other part describes the changing effect of air volumes by increasing f_h values with increased air contents. These two distinct behavior patterns may be explained by the nature of the model system used. A plateau may exist when void areas around the brick periphery are filled with air, but an air layer has not formed on the brick surface so heat transfer is maintained. Heating rate indices

Figure 3. Heating rate index as a function of included air; Lagarde retort, thin bricks.
(a, 115°C; b, 120°C; c, 125°C)

Figure 4. Heating rate index as a function of included air; Lagarde retort, thick bricks.
 (a, 115°C; b, 120°C; c, 125°C)

begin to increase once the outer void locations have been filled and a layer of air begins to form over the brick face. Differences between conditions may be due to the volumes of air at which a transition from one part to the other occurs. In conditions described as a horizontal line, the transition or break-point would occur at volumes of air larger than the maximum tested. Cases where f_h increases at very low air volumes demonstrate a very small or imperceptible plateau, so no apparent break-point exists.

Due to the nature of the data, comparisons between variables were performed in two ways. First, volumes of air at which a transition from the plateau region to an upward trend (if it exists) were determined. Secondly, comparisons were made between conditions where appreciable data existed in the upward section or part two of the model.

IV Break-point Values

1. Determination of Break-point

In all retorts, the f_h vs. included air relationships at 125°C and 65% steam or pressure level 1 exhibited a horizontal line. This may be expected since these conditions represent the highest retort pressure tested. Due to flexibility of the pouch, it has been shown to respond to theoretical pressure volume relationships (Nelson et al., 1956). It is clear from the data that conditions of 125°C and 65% steam (pressure level 1) limit pouch expansion so no restriction on heat transfer occurred in the range of air volumes tested.

A test on significance of the regression was performed to statistically determine if a horizontal line exists (Zar, 1974). A null hypothesis of slope equal to zero was tested using a Student's t-test.

Results of this analysis in Table 6 indicated that in all retorts and for each thickness all lines were essentially horizontal ($p>0.05$). Based on this finding, the horizontal line of 125°C and 65% steam/pressure level 1 was used as a baseline for means of comparison.

A Student's t -test was also used to establish if a difference exists between packaged and unpackaged bricks. Since no relationship exists between f_h as a function of included air for the baseline relationship these values were compared against bare brick f_h values. Results in Table 7 show that the two means differ ($p<0.05$) in five out of six cases. No significant difference was demonstrated in the positive flow retort with thin bricks. In all cases the mean f_h was larger for packaged bricks than their unpackaged counterparts. Differences may be attributed to an added resistance to heat transfer from the pouch and 10 mL of water added. Another possible reason may be that pouch edges extended outward and caused reduced media circulation between samples.

Comparisons between regression lines were performed using covariance analysis for all conditions in each retort against the respective baseline. Thick and thin bricks were analyzed separately because they had different magnitudes of baselines. Those conditions which displayed no significant difference from the baseline in either slope, level or overall ($p>0.05$) were considered as horizontal lines with no statistical evidence of a break-point. Conditions where a significant difference was determined in either slope or level ($p<0.05$) were considered to have an upward section (in all cases, either slope or level were higher).

TABLE 6.

Test for significance of slope of the baseline
125°C-65% steam with Student t-test.

H_0 : slope = 0.

Brick Thickness	Retort	Slope (min/mL)	t calc
Thin	Lagarde	0.012	0.0319 ns
	Positive flow steam/air	-0.0032	-0.0055 ns
	Positive flow water/air	0.0089	0.0515 ns
Thick	Lagarde	0.0041	0.0055 ns
	Positive flow steam/air	0.0056	0.0046 ns
	Positive flow water/air	0.0009	0.0015 ns

ns not significant ($p > 0.05$): accept H_0

TABLE 7.

Student t-test on f_h of unpackaged bricks
 compared to f_h values of baseline.
 $H_0: \mu_1 = \mu_2$

Brick	Retort	mean f_h , min		t calc.
		unpackaged	packaged	
Thin	Lagarde	6.08	6.83	6.24**
	Positive flow steam/air	6.42	6.71	1.91 ns
	Positive flow water/air	6.46	6.95	3.97**
Thick	Lagarde	17.78	19.74	7.58**
	Positive flow steam/air	19.24	19.49	2.56*
	Positive flow water/air	18.32	20.12	6.18**

ns not significant ($p > 0.05$)

* significant at $p < 0.05$: reject H_0

** significant at $p < 0.01$: reject H_0

The procedure used for determining points of transition or break-points may be described as covariance analysis on a moving regression frame. This concept was previously described by Tung et al. (1984a) for use in a different application. It was employed to determine the highest air volume at which data show no significant difference ($p > 0.05$) in either slope or level from the baseline. The moving frame was performed by beginning with the last ten consecutive data points of highest included air volumes. Ten data points were chosen because that amount allowed for desired sensitivity of the test, yet represented normal fluctuation in the data. Covariance analysis was performed at each stage as the frame moved in order of decreasing air contents until the line showed no significant difference, in all respects, from the baseline. Figure 5. schematically depicts how the analysis was performed. Frames in each condition were compared against the baseline from respective retorts. The highest air volume of the first frame exhibiting no significant difference ($p > 0.05$) represents the transition or breakpoint. The determined values are listed in Tables 8 and 9. An additional comparison was performed on all data up to the break-point as illustrated by the dashed line in Figure 5. In all cases, there was still no significant difference ($p > 0.05$) from the respective baselines. Air volumes up to the determined break-points were then considered to be the plateau region. As determined by the moving regression frame, if a larger air volume was included, the relationship became significantly different from the baseline. These analyses were performed on all conditions with evidence of a break-point. The same method was repeated consistently for all retorts, processing conditions and brick

ns not significant ($p>0.05$)
* significant at $p\leq 0.05$.

Figure 5. Moving regression frame performed with covariance analysis to determine break-points.

TABLE 8.

Break-point values of thin bricks determined by covariance analysis on a moving regression frame.

Retort	Temperature °C	Break-point Values (mL)		
		65% steam (1)	75% steam (2)	85% steam (3)
Positive flow steam/air	115	17.0	10.2	9.0
	120	29.5	18.5	13.6
	125	29.2	28.8	9.1
Positive flow water/air	115	29.2 ¹	24.8	15.9
	120	30.8 ¹	28.2	24.6
	125	30.4	34.3 ¹	20.2
Lagarde	115	29.8 ¹	20.3	11.6
	120	28.7 ¹	26.0	14.4
	125	31.7 ¹	31.8	<5.0 ²

¹ns to maximum air volume.

²sd to minimum air volume.

TABLE 9.

Break-point values of thick bricks determined by covariance analysis on a moving regression frame.

Retort	Temperature °C	Break-point Values (mL)		
		65% steam (1)	75% steam (2)	85% steam (3)
Positive flow steam/air	115	15.3	11.0	<5.0 ²
	120	31.8 ¹	28.9	9.3
	125	34.5	27.3	<5.0 ²
Positive flow water/air	115	32.3 ¹	13.2	<5.0 ²
	120	29.8 ¹	25.0	9.3
	125	31.8	35.0 ¹	13.3
Lagarde	115	31.6 ¹	16.4	<5.0 ²
	120	28.8 ¹	19.4	9.7
	125	30.3	30.7 ¹	<5.0 ²

¹ns to maximum air volume.

²sd to minimum air volume.

thicknesses. Since only one value of break-point may be determined, differences between values may not be statistically tested. Therefore, comparisons are made by differences in magnitude of each break-point.

2. Factors Affecting Break-Point Volumes

a. Steam Percentage or Pressure Level

Results shown in Tables 8 and 9 may be studied from the standpoint of differences in steam percentages or pressure level. By examination of values according to rows, the degree of overpressure may be compared for each variable of temperature, retort and brick thickness. In all cases, break-point volumes of air increased as the degree of overpressure increased up to 65% steam/pressure level 1 in this study. It is apparent that at higher degrees of overpressure, larger air volumes may be entrapped with no interference in heat transfer. Results from bare bricks demonstrated no significance ($p > 0.05$) in degree of overpressures tested. Therefore, the differences observed may be attributed to the influence of processing conditions on entrapped gases.

As previously explained, the test samples consisted of rectangular bricks packaged in flat pouches. Air entering the pouch could first accumulate around the thin edges of the bricks, then as volumes of air increase layers could form over the larger surfaces of the brick. Tables 8 and 9 indicate that almost all conditions with 65% steam/pressure (level 1) limit expansion so that entrapped gases remained only in void areas near the brick edges. Conditions of reduced overpressure allowed only small volumes of air to exist in peripheral brick areas because expansion during processing occurred to a larger degree. Above the break-point, a cushion of air may have accumulated between the large

faces of the brick and the pouch material, thereby impeding heat transfer to the centrally located thermocouple. An example in Table 8 of thin bricks processed in the Lagarde retort at 115°C displayed break-points of 29.8 mL (maximum air volume) at 65% steam, 20.3 mL for 75% and 11.6 mL for 85% steam. Thick bricks processed at 115°C and 85% steam (level 3) showed no evidence of a plateau section in all three retorts. Consequently, sufficient expansion may have occurred to interfere with heat transfer at small occluded air volumes (<5.0 mL) when processing with 30 kPa air overpressure.

Results of this study indicate that when processing with lower steam ratios (65% steam), heat transfer in pouches with large amounts of air will be maintained. Studies by Pflug and Borrero (1967) established that higher steam/air ratios provided a more effective medium because of increased enthalpy and heating efficiency. However, findings in these experiments suggest that a higher degree of overpressure will improve effectiveness of heating when large amounts of air are entrapped in the package. This study makes no predictions below 65% steam (level 1) as a limit exists where excessive air overpressure will reduce heating efficiency in the media.

Most previous studies dealing with residual gas have focussed on either pressure-volume relations to determine maximum allowable gas volumes to prevent pouch bursting (Whitaker, 1971) or the interference by entrapped air on heat transfer at a single processing condition. In a study by Evans (1977), heating rate indices were evaluated for pouches of variable air content processed at two levels of overpressure. His results showed an increase in f_h with high air volumes (50 mL), yet no

effect was contributed by overpressure even when pouches were restrained (as compared to constrained). Conditions studied were similar to 120°C and 125°C at pressures corresponding to 75% steam and 85% steam. Differences from this study may be due to the fact that an overpressure representative of 65% steam was not tested and higher air volumes were evaluated. A study by Pereira (1980) evaluated various degrees of vacuum on pouches with two overpressure levels. The reported results were inconsistent; however, for one thickness (thinnest package) with a low vacuum setting (may assume high air) a shorter sterilization time was required when a higher degree of overpressure was used. This finding is similar to results in this study where higher overpressure processing limited gas expansion and maintained heat transfer.

b. Temperature

Effects of temperature may be compared by studying the tables in columns for each section. At conditions of 65 and 75% steam (pressure levels 1 and 2), permissible air volumes increase with increasing temperature. In some cases, for example at 65% steam (level 1), it reached the maximum air level tested and remained as temperature increased. This trend may be explained by larger saturation vapor pressures at high temperatures. This results in a higher retort pressure setting when employing the same steam percentage. The same principle is employed for UHT processing of flexible pouches in Japan (Tsutsumi, 1979a). Superheated steam at high temperatures provide high saturated vapor pressure to adequately process pouches in pure steam (for heating only) while preventing pouch bursting.

At conditions of 85% steam (pressure level 3) volumes deviate

from the pattern displaying lower break-point values at 125°C. A possible explanation of this occurrence in trials performed in the pilot scale retort may be due to low rotameter settings at conditions of 85% steam (pressure level 3). Although media flow rates were constant, the amounts of air passing through the system were lower. Media circulation may have been insufficient and resulted in poor heat transfer. A similar situation, however, was found in the Lagarde. Media flow rates are very high in this system due to the turbo fan circulation mechanism.

c. Brick Thickness

Although the two brick thicknesses have much different magnitudes of baseline f_h values, volumes of air which demonstrate no significant difference from the baseline may be compared by studying values in Table 8 and Table 9. In almost all cases where a break-point exists, the maximum air volume of the horizontal section is higher for thin than for thick bricks. For example, with water/air processing at pressure levels 2 and 3 at 115°C and level 3 at 120°C, there is approximately a 10-15 mL difference in volume of air forming the plateau region between thicknesses. This magnitude of difference occurs in some other conditions, but not every case.

Variations in the volume of air at which no influence on heating rate occurs may be a reflection of the pouch size in relation to the brick volume. Since similar pouches were used to package both brick sizes, there would be a difference in the potential volume of space available around the brick edges. Consequently, more air could accumulate in surrounding spaces before residing over the brick face when thin bricks were packaged compared to pouches containing thick

bricks.

d. Media Type: Steam/Air and Water/Air

Modification of the vertical positive flow retort to facilitate water/air processing enabled studying different processing media within the same retort. In order to compare break-point values of conditions using the media types, a covariance analysis was performed on the baselines. No significant difference ($p > 0.05$) was found between baselines of water/air media and steam/air in the vertical positive flow retort (see Table 10) for either thick or thin bricks. Although a significant difference ($p < 0.05$) was found between retorts for unpackaged thick bricks, it is not apparent when comparing baselines. This may be attributable to larger variability in data when air was included in the package.

Break-point values for these systems in Tables 8 and 9 may be studied. It was apparent that superheated water with air overpressure provided processing media which allowed larger volumes of air to be included in the sample pouches with no increase in f_h values. Data for thin bricks reflected this occurrence strongly; at 115°C for each overpressure level, values for water/air processing ranged from 7-14 mL higher than for steam/air. A difference between media types was demonstrated by both brick thicknesses at 115°C and 65% steam (pressure level 1). When processing with steam/air a break-point was exhibited at 15.3 mL (thin) and 17.0 mL (thick) of included air, however, there was no evidence of a break-point with similar processing conditions when employing water/air media.

These studies indicated that when using water/air processes,

heating rate indices were affected to a lesser degree by included air than when steam/air mixtures were used. Improved heat transfer using superheated water with air overpressure may be due to an increase of pressure due to a hydrostatic head. A water depth of approximately 1 meter in the vessel would contribute 10 kPa pressure. Bare brick analysis in the case of thick bricks demonstrated that water/air media may be more effective independent of air volumes. Other researchers have studied media types with similar results. Yamano (1976) compared heating rates of pouches in steam/air and water/air media. Although long come-up times were associated with water processes, when the media reached desired temperatures, heating rates were faster than with steam/air mixtures. Pflug and Borrero (1967) recommended use of water/air over steam/air media based on simplicity of use and higher enthalpies associated with water/air mixtures per unit volume. Tsutsumi (1979b) stated that water/air processes were commonly used in Japan when pouches contained larger amounts of included air.

e. Retorts: Lagarde and Positive Flow Steam/Air

Steam/air media with similar processing conditions were utilized in both the Lagarde and vertical positive flow systems. Analysis of covariance was performed on baseline conditions (125°C, 65% steam) of each retort. No significant difference ($p > 0.05$) was found between the relationships in each brick thickness (Table 10). Although a significant difference was found between retorts for both bricks unpackaged, this was not apparent when comparing baselines. This may be attributable to larger variabilities in data when air was included in the packages. Values in Tables 8 and 9 may be compared for these

TABLE 10

Covariance analysis results comparing baselines
between retort systems tested.

Brick Thickness	Comparison	F Ratio		
		Test of Slope	Test of Slope	Overall
Thin	Pos. flow s/a vs. Pos. flow w/a	0.69 ns	3.51 ns	2.10 ns
	Pos. flow s/a vs. Lagarde	0.44 ns	0.37 ns	0.40 ns
	Pos. flow w/a vs. Lagarde	0.08 ns	0.21 ns	1.08 ns
Thick	Pos. flow s/a vs. Pos. flow w/a	3.51 ns	2.91 ns	3.30 ns
	Pos. flow s/a vs. Lagarde	1.57 ns	0.59 ns	1.08 ns
	Pos. flow w/a vs. Lagarde	0.31 ns	1.09 ns	0.54 ns

ns - not significant ($p > 0.05$)

systems. Larger volumes of air comprise the plateau region for the Lagarde compared to the vertical positive flow system. This occurrence was demonstrated in both brick thicknesses. Results from conditions of 115°C and 65% steam exemplify the difference. Sufficient conditions exist in the Lagarde retort so no interference in heating rate was apparent at the maximum volumes of air added. However, a break-point was detected at 17.0 mL (thin bricks) or 15.3 mL (thick bricks) when processing with similar retort conditions in the vertical positive flow retort. Other conditions exhibit the same trends.

These results indicate that systems used for processing may demonstrate an effect on the heating rate of products containing residual gases. Since similar media compositions were utilized, differences may be due to the effectiveness of circulation. In Lagarde retorts a horizontal air flow pattern is ensured by circulation with a turbo fan causing media to flow parallel across the brick surface. Media flow in the vertical positive flow retort is in an upward direction, perpendicular to the brick plane. Therefore, circulation may be hindered by bricks on the bottom layers.

3. Importance of Plateau Region

Study of the data showed heating rate indices as a function of air entrapped in packaged bricks to consist of either one or two sections. One part was essentially a horizontal line, the other exhibited an increase in f_h with increasing air contents. This two part behavior may be explained by the rigid nature of teflon bricks forming void areas in the brick periphery. Care must be taken in extrapolating results to a

food system. A food product of softer and more pliable form would fill out to the edges and air would accumulate on top. Consequently, data from Tables 8 and 9 may not directly be extracted for implementation as quality control specifications (on the basis of heat transfer) without knowledge concerning the behavior of air with pouches containing foods. The results do suggest that included air limits may be set at higher levels and not contribute a limiting factor to process times when high overpressures are used and adequate circulation is ensured. Any changes in quality control levels must be tested on a food product, and other deteriorative implications of entrapped gases must be evaluated. Also in regard to specific quality control levels, conditions of 115°C and 85% steam (level 3) may be a concern because air volumes of less than 10 mL displayed an interference with heat transfer.

V Air Volumes Above the Break-Point

The previously described moving regression frame determined values of included air at which a transition or break-point occurred (if present) from a horizontal line, to one portraying an increase in heating rate indices with higher air volumes. Work by previous researchers have shown a relationship of this nature where included air resulted in an insulating effect on heat transfer. Evans (1977) reported increases in f_h values, Huerta-Espinosa (1981) described the insulating effect by increased times required for sterilization and Berry and Kohnhorst (1983) found a similar behavior evaluated by decreasing sterilization values or F_0 .

Experimental variables may be compared by covariance analysis on the data existing beyond the break-point describing the increasing

trend. A simple arithmetic linear relationship was found to best describe the data. A large standard error of estimate may be expected as large variations exist in f_h values. Only some conditions may be compared since many relationships exhibited only horizontal sections and others showed increases at high air volumes, beyond which insufficient information was available for a reliable regression line. Therefore, comparisons of equations were focussed on those conditions with appreciable data in the sections above the break-point. Slopes of the lines were parameters of interest since differing ranges of air volumes were used and intercepts were inevitably different.

1. Factors Affecting the Second Section

- a. Brick Thickness

In the previous section it was determined that thin bricks could accommodate larger volumes of air than thick bricks without impeding heat transfer. The degree of increase beyond the plateau region was compared. Retort conditions of 115°C and 85% steam (pressure level 3) provided a large amount of data beyond the break-point, so the relationships were used for comparison. As indicated in Table 11, no significant difference ($p > 0.05$) was found between slopes in the relationship of heating rate index as a function of air volume for thin and thick bricks. It may be assumed that once a critical air volume was reached and heating rates began to be affected, additional air volumes added to the pouch increased f_h with the same severity for both brick thicknesses. Consequently, a cushion of air of similar thickness was formed (past critical volumes) for each additional volume of air added.

TABLE 11

Covariance analysis on f_h vs. included air, values above break-point;
comparison of brick thickness, 115°C-85% steam (pressure level 3)

Retort	Brick Thickness	Slope (mL/min)	Comparison	Test of Slope F Ratio
Lagarde	Thin Thick	0.396 0.254	Thin vs. Thick	2.24 ns
Positive flow steam/air	Thin Thick	0.091 0.104	Thin vs. Thick	1.33 ns
Positive flow water/air	Thin Thick	0.192 0.261	Thin vs. Thick	0.21 ns

ns not significant ($p > 0.05$)

It was apparent in Tables 8 and 9 that break-point volumes were larger for thin compared to thick bricks. Further comparisons of variables were performed on data of thick bricks.

b. Steam Percentage/Pressure Level

Conditions of variable steam percentages (pressure levels) were compared at 115°C for thick bricks in all three systems. Although 115°C was used since it contained the lowest break-point volumes of air, in two systems a horizontal line or plateau section only was displayed with 65% steam (level 1). If no break-points were apparent, the relationships were not used for comparison. Results are reported in Table 12. In one of the three systems (Lagarde), a significant difference ($p < 0.05$) was found between slopes of 85 and 75% steam, the former having a larger slope than the latter. The relationships are illustrated in Figure 6. This finding supports evidence that higher degrees of overpressure limit expansion of residual gases. The larger slope resulting from processing with 85% steam may be attributed to a lower degree of overpressure allowing expansion of gases. Therefore, the insulation effect was more severe as additional air was included.

Comparisons of steam percentages in other systems did not reflect these findings statistically since no differences ($p > 0.05$) were found. However, slopes were largest for 85% steam then decreased for 75 and 65% steam, respectively. This indicated that higher degrees of overpressure may have limited expansion to a larger degree to result in a more gradual increase of heating rate. However, it must be noted there was no significant difference. The vertical positive flow retort employing water processing with air overpressure also demonstrated no

TABLE 12

Covariance analysis on f_h vs. included air, values above break-point;
comparison of pressure level, at 115°C.

Retort	% Steam/ Pressure Level	Slope (mL/min)	Comparison	Test of Slope F Ratio
Lagarde	85 75	0.271 0.540	85 vs. 75	4.84*
Positive flow water/air	3 2	0.145 0.104	3 vs. 2	0.54 ns
Positive flow steam/air	85 75 65	0.260 0.178 0.088	85 vs. 75 75 vs. 65 85 vs. 65	1.14 ns 0.83 ns 4.02 ns

ns not significant ($p > 0.05$)

* significant ($p < 0.05$)

Figure 6. Heating rate index vs. included air for comparison of pressure level; thick bricks processed at 115°C in the Lagarde retort.

significant difference ($p>0.05$) between slopes at different pressure levels. A similar trend was evident since the slope at pressure level 3 was slightly steeper than at pressure level 2 (corresponding to 85 and 75% steam, respectively). Pressure level 1 exhibited a horizontal line with no evidence of an upward trend.

c. Temperature

The effects of temperature on the f_h vs. included air function were compared at a constant degree of overpressure. Conditions at 85% steam (pressure level 3) were tested because more information was available for the second equation in the model. Table 13 shows the results of the covariance analysis. In the Lagarde and positive flow water/air retorts there was no significant difference ($p>0.05$) between slopes of the lines for all three temperatures. This would indicate that although each temperature may allow for differing volumes of air to be included without demonstrating an effect on heat transfer, the severity of increases above the critical value showed no significant difference. In the vertical positive flow system employing steam/air mixtures a significant difference ($p<0.05$) was found between slopes of the relationships for 115 and 125°C, the former being greater than the latter. The slope of 120°C was intermediate to 115°C and 125°C showing no significance ($p>0.05$) from either value. Figure 7 depicts the relationships which exhibited a significant difference. A larger slope at a lower temperature supported results found in the previous section describing break-point values. Lower saturation vapor pressures associated with low temperatures could cause expansion of entrapped gases to a larger degree, resulting in a more acute interference in heat transfer as

TABLE 13

Covariance analysis on f_h vs. included air, values above break-point;
comparison of temperature at 85% steam (pressure level 3).

Retort	Temperature (°C)	Slope (min/mL)	Comparison	Test of Slope F Ratio
Lagarde	115	0.271	115 vs. 120	0.71 ns
	120	0.209	120 vs. 125	0.09 ns
	125	0.236	115 vs. 125	0.16 ns
Positive flow water/air	115	0.145	115 vs. 120	0.07 ns
	120	0.140	120 vs. 125	3.15 ns
	125	0.056	115 vs. 125	3.37 ns
Positive flow water/air	115	0.260	115 vs. 120	0.86 ns
	120	0.198	120 vs. 125	0.27 ns
	125	0.174	115 vs. 125	5.40 *

ns not significant ($p > 0.05$)

* significant at $p < 0.05$

Figure 7. Heating rate indices vs. included air for comparison of temperature; thick bricks processed at 85% steam in positive flow retort.

displayed by the larger slope.

d. Media Type: Steam/Air and Water/Air

Comparisons between steam/air and water/air media in the vertical positive flow retort system were made on equations describing data beyond critical values. Conditions of 75% steam (pressure level 2) and 85% steam (pressure level 3) at 115°C and 120°C were studied since there was substantial information for comparison. Conditions of 65% steam demonstrated a plateau section only for water/air processing; in these cases no comparisons were made. In Table 14 are results from the test of slopes by covariance analysis. In one condition, 115°C and 85% steam, a significant difference ($p < 0.05$) was found between media types. Other conditions demonstrated no significant difference ($p > 0.05$). It may be expected that 115°C and pressure level 3 would show differences to a larger degree since they represented retort conditions of the lowest pressure. Data from these relationships (Figure 8) provided the best lines for comparisons since there were no apparent plateaus. The slope for steam/air was significantly higher than for water/air which supports evidence found when comparing break-point values. A larger slope found with steam/air processing reflects that steam/air mixtures may provide a less efficient heating medium resulting in greater degradation of heat transfer with additional amounts included air. It may be possible that hydrostatic pressure in the water/air system contributed to limiting the expansion of non-condensable gases, thereby resulting in a lower slope.

Other conditions tested demonstrated no significant difference ($p > 0.05$); however, in all cases slopes were larger for steam/air pro-

TABLE 14

Covariance analysis on f_h vs. included air, values above break-point;
comparison of media type.

Processing Conditions	Media Type	Slope (min/mL)	Comparison	Test of Slope F Ratio
115°C-75% steam (2)	Steam/air	0.189	s/a vs. w/a	0.12 ns
	Water/air	0.151		
115°C-85% steam (3)	Steam/air	0.260	s/a vs. w/a	13.8 *
	Water/air	0.104		
120°C-75% steam (2)	Steam/air	0.179	s/a vs. w/a	0.15 ns
	Water/air	0.050		
120°C-85% steam (3)	Steam/air	0.198	s/a vs. w/a	1.07 ns
	Water/air	0.140		

ns not significant ($p > 0.05$)

* significant at $p < 0.05$

Figure 8. Heating rate index vs. included air for comparison of media type; thick bricks processed at 115°C and 85% steam (3).

cesses. Although a trend was evident, the slopes were not statistically different. Combining this information with results from critical volumes which displayed no significant difference from a baseline, it may be assumed that water/air processes show an improvement over steam/air processes with regard to the effects of entrapped gases on heat transfer.

e. Retort Design: Lagarde and Positive Flow Steam/Air

Similar to previous comparisons, there were appreciable data beyond the critical volume. Results from Table 15 indicated no significant difference ($p > 0.05$) between slopes of the relationships tested. Values of slopes revealed no consistencies as some values were larger in the Lagarde and some in the vertical positive flow retort. Figure 9 illustrates an example of these relationships. From results on break-point values of the plateau regions, it was evident that larger volumes of air could be included without affecting heat transfer when processing in the Lagarde retort. Forced circulation by the turbo fan and media flow parallel to the brick surface may be responsible for this difference. It may be expected that slopes of the increasing relationship would be larger (more severe) for the vertical positive flow system; however, this was not the case. In some cases larger slopes were found when processing in the Lagarde, which may be due to the absence of an additional spacer layer that was inherent in racks used with the vertical positive flow retort. As air volumes increased beyond the critical point, rack clearance above the pouch would be reduced. In the Lagarde retort an adequate clearance was necessary to ensure circulation, whereas in the positive flow retort a spacer layer ensured circulation even

TABLE 15

Covariance analysis on f_h vs. included air, values above break-point;
comparison of steam/air retorts.

Processing Conditions	Retort	Slope (min/mL)	Comparison	Test of Slope F Ratio
115°C-75% steam	Lagarde	0.053	Lag. vs. Pos.	1.45 ns
	Pos. flow	0.189		
115°C-85% steam	Lagarde	0.271	Lag. vs. Pos.	0.04 ns
	Pos. flow	0.261		
120°C-75% steam	Lagarde	0.138	Lag. vs. Pos.	.007 ns
	Pos. flow	0.180		
120°C-85% steam	Lagarde	0.209	Lag. vs. Pos.	.024 ns
	Pos. flow	0.198		

ns not significant ($p > 0.05$)

Figure 9. Heating rate index vs. included air for comparison of steam/air retorts; thick bricks processed at 115°C and 85% steam.

if clearance space was diminished. Other researchers have determined racking configurations to be an important factor in processing. Evans (1977) compared restrained and constrained racking designs by evaluating heating rate indices of pouches containing residual gases. Constraint of pouches reduced the extent of interference by entrapped gases on heat penetration. Ramaswamy (1983) studied vertical and horizontal orientation with constrained and unconstrained rack types. Results showed that processing with unconstrained racking in the horizontal orientation reduced heat transfer rates. From this study, it was evident that if "unconstrained" racking was employed, clearance above the pouch may be reduced when large volumes of air are entrapped. To ensure proper circulation, adequate flow channels should be provided between sample layers.

VI Prediction of Pouch Expansion

Previous sections have dealt with the relationship of f_h as a function of residual gases measured at conditions of standard temperature and pressure (STP). These relationships were valuable in a practical sense to determine heating rates of products since similar air volume measurements are performed in quality control programs. However, variables may be studied by predicting the volume of air which would prevail at retort conditions. Analysis of variance performed on bare bricks showed no significant differences in variables of temperature and pressure level. When air was included in a pouch, differences were attributed to an interaction between entrapped air and retort conditions of temperature and pressure. Predicted volumes of air may account for

any interaction so all processing conditions in each system may be combined.

A major study on pressure-volume relations by Davis et al. (1960) revealed that, due to flexible nature of the pouch, ideal gas theories applied. Pressures within the pouch arose from vapor pressure of water in food, air pressure in the headspace or released from the product and thermal expansion of the food itself. Whitaker (1977) derived equations based on Boyle's law and Dalton's law of partial pressures in order to find a total overriding pressure necessary to limit expansion beyond a maximum volume (determined by confinement). A specific example was cited where volume expansion of packaged peas in brine was calculated when no air was entrapped and when air was included. Studies by Yamano (1976) utilized the same concepts. With water and air contained in a pouch, expansion was primarily caused by air pressure changes within the headspace. Expansion ratios (expansion headspace equal to pouch expansion) were determined for different retort conditions. Assuming a maximum expansion ratio to preclude pouch bursting, a safe overriding air pressure of 20-30 kPa was determined.

1. Expansion Factors

Pressure volume relationships were used to predict volumes of air existing at retort conditions. In a system of brick, water and air, expansion of all components must be quantified. Expansion of water was considered negligible or decreasing, and since only 10 mL of water was added, any changes were small. Brick expansion may be estimated from the characteristics of teflon which has a linear thermal expansion factor (α of $10^{-5}/C^{\circ}$) as listed in Table 1. Volume expansion (β) was

calculated by the following relationship:

$$V_t = V_o (1 + \beta \Delta t) \quad \dots (12)$$

where V_o is original volume, V_t is volume at retort conditions, and Δt denotes temperature difference. Considering even the large brick and a temperature change of $100C^o$, volume expansion was insignificant. Expansion due to entrapped air was therefore the most important component when considering the potential effect on heating rate. The relationship describing expansion ratios volumes is as follows:

$$\text{Expansion Factor (EF)} = \frac{V_2}{V_1} = \frac{P_1 T_2}{T_1 P_a} \quad \dots (13)$$

where P_1 and T_1 are room pressure and temperature (in absolute), T_2 is retort temperature (in absolute) and P_a indicates pressure exhibited by air within the pouch. Total pressure within the pouch was assumed to be equal to retort pressure because of the flexible nature of the film. Since 10 mL of water was added to the pouch, vapor pressure will contribute to the total internal pressure. Dalton's law of partial pressures was applied to calculate pressure due to air within the system, P_a , from the retort pressure minus saturation pressure of water at retort temperature. An expansion factor of V_2/V_1 (the ratio of volume of air at retort conditions to volume of air measured) may be found. Expansion factors were calculated for all retort conditions tested. An example calculation at 115^oC -75% steam is as follows:

P_1 = atmospheric pressure 100 kPa.

P_a = retort pressure - saturation vapor pressure at 115°C
= 56.3 kPa.

T_2 = retort temperature = 388°K.

T_1 = room temperature = 298°K.

$$\text{Expansion factor, } \frac{P_1 T_2}{T_1 P_a} = 2.31 \quad \dots (14)$$

This expansion factor indicates that for each mL of air added to the pouch it will expand 2.31 times when reaching retort conditions. Other values of expansion factors for retort conditions studied are in Table 16.

Factors tabulated for each condition provide information aiding explanation of findings in previous sections. The magnitude of expansion factors reflects volumes of air included in plateau regions. It is apparent in Table 16 that a decrease in overpressure from 65% steam to 85% steam results in an increase in expansion factors. Additionally, a decrease in expansion factor was calculated as temperatures increase (with a constant degree of overpressure). It may be noted that the baseline relationship of 125°C and 65% steam (level 1) used for comparisons to determine the breakpoint has an expansion factor close to one (1.07). As previously discussed, there was an increase of maximum volumes displaying no significant difference from the baseline, as the degree of overpressure increased. Similarly, increasing temperature at the same overpressure was characterized by larger plateau regions. Results showed that when expansion factors were small, large volumes of

TABLE 16

Theoretical expansion factors for conditions studied.

Temperature, °C	Steam (%)		
	65	75	85
115	1.43	2.31	4.36
120	1.25	1.99	3.76
125	1.07	1.73	3.26

air could be included within the pouch without interfering with heat transfer.

Measured volumes of air may be adjusted by multiplying by respective expansion factors to determine the corresponding volumes of air prevailing during processing. Retort temperature and pressure were set by control mechanisms as previously described; however, in many instances actual conditions may have slight deviations from the target. To correct for these differences, all volumes of air were adjusted based on expansion factors calculated from actual retort conditions. Figures 10 and 11 illustrate relationships between the log of heating rate indices and volume of air adjusted with expansion factors (on an arithmetic scale). Uneven spacing of data points along the abscissa was due to differences in expansion factors. Factors for conditions of 65% steam (level 1) varied from approximately 1.07 for 125°C to 1.43 at 115°C; therefore, values were spread out over lower adjusted volumes. Expansion factors of 1.73 (125°C) to 2.31 (115°C) were determined for 75% steam (pressure level 2) with data ranging from low to medium adjusted volumes. Conditions of lower overpressure with pressure level 3 (85% equivalent) were represented by expansion factors ranging from 3.26 (125°C) to 4.36 (115°C) so data were spread across the entire abscissa from low to high adjusted air contents.

2. Comparison of Adjusted Volume Relationships

A semi-logarithmic relationship clearly indicated an increase in f_h with increasing adjusted volumes of air. The plateau section as demonstrated with included air volumes were no longer apparent because

Figure 10. Heating rate index functions of adjusted air volumes for all conditions; thin bricks. (a, Lagarde; b, Positive Flow Steam/Air; c, Positive Flow Water/Air).

Figure 11. Heating rate index functions of adjusted air volumes for all conditions; thick bricks. (a, Lagarde; b, Positive Flow Steam/Air; c, Positive flow Water/Air).

those values remained at lower ranges of the adjusted volume abscissa. Covariance analysis was performed to compare retort and media systems employed in these experiments. Table 17 shows results of the analysis and slopes describing each relationship. A significant difference ($p \leq 0.01$) was found between all three systems for both thicknesses. From values of the slopes and observation of Figures 10 and 11, results indicate that processing with steam/air media utilizing the vertical positive flow retort, displays a more severe effect of residual gases on heating rate than demonstrated by processing in the Lagarde. Above all, water with air overpressure media was less sensitive to increasing air compared to both steam/air systems, as demonstrated by a significantly lower slope. The results were similar for each brick thickness and concur with findings from unadjusted air volumes. Differences in steam/air systems may be attributable to the turbo fan driven media circulation and flow patterns parallel to pouch position in the Lagarde. This system provides more rapid heating than the vertical positive flow retort. Higher heat transfer coefficients for water/air compared to steam/air may account for a more gradual increase in $\log f_h$ as a function of adjusted air volume. Another plausible explanation may be due to an increase in pressure from hydrostatic forces in the system. The results were contrary to findings by Milleville (1980) where a buoyant effect of the residual air bubble within the pouch was expected to reduce heat transfer. However, observations of the buoyant effect of air inside the pouch were performed at atmospheric pressure.

The adjusted air volume approach may prove useful in predicting heating rate indices of pouches containing residual gases when

TABLE 17

Covariance analysis on $\log f_h$ vs. adjusted air, all data;
comparison of systems tested.

Brick Thickness	Retort System	Slope (mL^{-1}) $\times (10^{-3})$	Comparison	Test of Slope F Ratio
Thin	Lagarde	2.32	Lag. vs. s/a	10.6 **
	Positive flow steam/air (s/a)	2.89	Lag. vs. w/a	99.2 **
	Positive flow water/air (w/a)	0.84	s/a vs. w/a	203 **
Thick	Lagarde	1.18	Lag. vs. s/a	8.13 **
	Positive flow steam/air	1.46	Lag. vs. w/a	22.5 **
	Positive flow water/air	0.77	s/a vs. w/a	70.4 **

** significant at $p < 0.01$

processing within the range of conditions studied. An estimate of expanded air volumes that will prevail at retort conditions can be calculated and heating rates may be determined. The model system used, simulating a food with water activity (a_w) equal to one, represents the most extreme case of pouch expansion. Many products have a slightly lower water activity and will exhibit a lower partial pressure due to water vapor. Expansion factors may be calculated to reflect these differences. For example, an expansion factor when $a_w = 1.0$ for 115°C and 85% steam is 4.36; compared to a calculated factor of 3.40 for a food of $a_w = .95$ at identical retort conditions. Quite a substantial difference exists, so water activity of the food processed must be considered an important factor when evaluating effects of residual gases on heating behavior.

Another practical application of this approach may aid when processing products such as bakery foods or batters in retort pouches. These goods require leavening and inclusion of non-condensable gases (usually CO_2) is essential for texture. Control of processing conditions is important to achieve a balance between allowing the product to rise, but preventing the pouch from bursting. Leavening, setting of structure and sterilization are the three critical factors of concern. Andres and Duxbury (1972) developed differential pressure sensing and controlling methods to achieve proper product texture and maintain pouch seal integrity during retort processing. They did not, however, provide details of their developments.

VII Other Process Parameters

As previously described, thermocouples were centrally located within the brick. When volumes of entrapped air form a layer on the surface, the coldest heating point would shift from the brick midplane. Temperature sensor placement errors were studied by Peterson and Adams (1985). Their results, based on determined process times, show that position of the thermocouple was not critical within a range of positions around the center of the slab. It was also evident that the position error allowed without affecting process times was greater for thick slabs. Measurement of f_h was not highly sensitive to thermocouple location within the sample, so this was not a great concern in experimentation. However, the lag factor j (a measure of the ratio of g value as estimated from a straightline portion of the heating curve to actual g value at steam on), is highly sensitive to thermocouple location. Berry and Kornhorst (1983) described coldest heating locations within pouches containing large amounts of air. It was found that with more than 50 mL of air in an institutional size pouch, the slowest heating point measured $2/3$ of the maximum pouch thickness. A similar testing must be performed for samples containing air in this experiment. Lag factors can then be evaluated in order to compare sterilization values of processes or time required to achieve a desired sterilization of pouches containing large amounts of residual gases.

CONCLUSIONS

The relationship of heating rate index as a function of included air may be described as consisting of either one or two essentially linear segments. One segment formed a plateau region where there was no change in f_h with increasing volumes of included air. The second section, apparent in some conditions only, exhibited an increase in f_h as a function of increasing air volumes.

Increased overpressure levels and temperatures (with the exception of 125°C and 85% steam) allowed for larger volumes of air to be included in the plateau region. Air overpressure above 80 kPa allowed up to 35 mL to be included with no detrimental effect on heating rates. Conversely, even small amounts of entrapped air (<5 mL) degraded heat transfer when processing with 30 kPa air overpressure. Increasing overpressure and temperature were also shown to display decreasing slopes in the second linear section where an increase in f_h is seen as a function of increasing air volumes.

Processing with water/air media allowed larger volumes of air to be included in the plateau region compared to steam/air. Both media were evaluated in a positive flow retort. As well, steeper slopes of the second segment were demonstrated with steam/air media indicating a more severe detrimental effect of entrapped air on heat transfer. Studies with retort types demonstrated that when processing in the Lagarde retort, larger volumes of air could be entrapped without affecting heat transfer than with the positive flow retort. No significance was seen in slopes of the second section.

Expansion factors for processing conditions were determined from

ideal gas laws. The factors were used to adjust air volumes for expansion that occurs at retort conditions. A linear relationship was found between the logarithm of heating rate index and adjusted air volumes on an arithmetic axis. Comparisons of slopes showed significant differences; water/air processes in the positive flow retort being of the least slope, the Lagarde and positive flow steam/air processes had increasing slopes, respectively.

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APPENDIX I

Non-destructive Air Measurement

Archimedes Principle:

$$B - Mg = \rho V_o g - \rho_o V_o g = (\rho - \rho_o) V_o g$$

where

B	=	buoyant force (upward)
Mg	=	weight of object (force downward)
ρ	=	density of surrounding fluid
ρ_o	=	density of object
V_o	=	volume of object
g	=	gravitational field

Applied to non-destructive air measurement

$$Mg - B = \rho_o V_o g - \rho V_o g = (\rho_o - \rho) V_o g$$

1. Before air addition

$$W_{wb} = M_b g - B_1 = (\rho_b - \rho_w) V_b g$$

where

W_{wb}	=	weight of brick in water
M_b	=	mass of brick
B_1	=	buoyant force (before air added)
ρ_b	=	density of brick
V_b	=	volume of brick

2. After air addition

$$W_{wb+a} = M_{b+a} g - B_2 = (\rho_{b+a} - \rho_w) (V_b + V_a) g$$

where

W_{wb+a}	=	weight of brick and air in water
M_{b+a}	=	mass of brick and air
B_2	=	buoyant force (after air added)
ρ_{b+a}	=	density of brick and air
V_a	=	volume of air

Expand these equations.

From Equation 1:

$$\begin{aligned}W_{wb} &= \rho_b V_b g - \rho_w V_b g \\&= M_b g - \rho_w V_b g \\M_b g &= W_{wb} + \rho_w V_b g\end{aligned}$$

From Equation 2:

$$\begin{aligned}W_{wb+a} &= \rho_{b+a} (V_b + V_a) g - \rho_w (V_b + V_a) g \\&= M_{b+a} g - \rho_w (V_b + V_a) g \\M_{b+a} g &= W_{wb+a} + \rho_w (V_b + V_a) g\end{aligned}$$

Assuming mass of air negligible, then $M_{b+a} = M_b$

$$W_{wb} + \rho_w V_b g = W_{wb+a} + \rho_w (V_b + V_a) g$$

Rearrange to:

$$\begin{aligned}W_{wb} - W_{wb+a} &= \rho_w (V_b + V_a) g - \rho_w V_b g \\&= \rho_w V_b g + \rho_w V_a g - \rho_w V_b g \\\Delta m g &= \rho_w V_a g \\\Delta m &= \rho_w V_a\end{aligned}$$

Assuming density of water at 25°C = 1.0 g/mL, for each mL of air added, the mass of the brick and air will be reduced by 1.0 g.

APPENDIX II

Back Calculations for Combination of Destructive and Non-destructive Air Measurements

1. Leakage of air during processing, mL:

$$= (B-A) \text{ 1.0 mL/g}$$

2. Non-destructive volume, post processing, mL:

$$= (I-A) \text{ 1.0 mL/g}$$

3. Initial volume of air (before air addition), mL:

$$= V - (I-A) \text{ 1.0 mL/g}$$

4. Projected weight of pouch containing no air, g:

$$= A + V \text{ 1.0 g/mL}$$

where: V = corrected destructive air measurement (mL)

I = first non-destructive air reading (g)
(prior to air addition)

B = second non-destructive air reading (g)
(before processing)

A = third non-destructive air reading (g)
(after processing)

APPENDIX IIIa

Heating rate index vs. included air relationship for thin bricks in positive flow steam/air retort (a, 115°C; b, 120°C; c, 125°C).

APPENDIX IIIb

Heating rate index vs. included air relationship for thick bricks in positive flow steam/air retort (a, 115°C; b, 120°C; c, 125°C).

APPENDIX IIIc

Heating rate index vs. included air relationship for thin bricks in positive flow water/air retort (a, 115°C; b, 120°C; c, 125°C).

APPENDIX IIIId

Heating rate index vs. included air relationship for thick bricks in positive flow water/air retort (a, 115°C; b, 120°C; c, 125°C).