



TECHNICAL / ENGINEERING SECTION

RESEARCH • METHODS • TESTING

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Integrity of food packages

Increasing use of sterilizable pouches prompts Army researchers to investigate their relative resistance to bacterial and extractive contamination. By J. W. Szczeblowski and F. J. Rubinate†*

Emphasis on quality and reliability of package performance has become a highlight in the field of foods. The 1963 botulism cases have caused many to question the integrity of packaged foods. The high standards set for cans have been impaired by the tuna incident and plastics packaging by the smoked-fish incident. As a result, the Food & Drug Administration has announced that inspectors will be checking to insure the soundness of containers. We can be certain that they will also be devoting more attention to newly developed concepts.

Boil-in-bag packaging of foods, introduced in the '50s, and, more recently, the sterilizable pouch (Figure 1) provide examples of some of these new ideas. Flexible packages for foods, whether they be frozen or processed by retorting, by aseptic methods or by radiation, have very similar factors that are of major importance related to the integrity of the flexible package. These are sealability, durability, extractives, microbial and insect penetration, shelf life and acceptance. Importance of these is measured variably, depending on what is considered of consequence. Except for extractives, standards have not been established in these areas for food products and are presently regulated by individual requirements only.

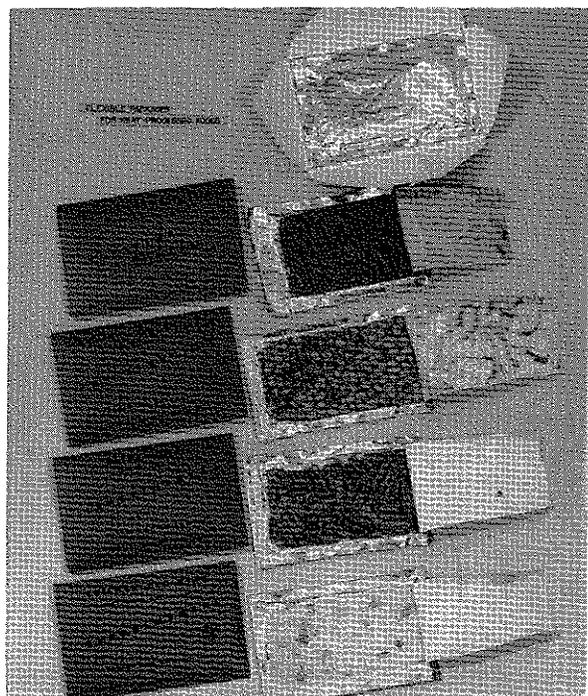
Because of the advantages and benefits the sterilizable pouch possesses, the military has been spearheading the development of the sterilizable pouch with the interest of replacing the metal can (Figure 2) in combat rations (1)¹. A number of commercial packers have expressed considerable interest in this concept of packaging, yet there is only one who is commercially producing tomato and fruit products in a sterilizable pouch (2). We sense that other packers have questioned the integrity of the package

and would prefer to have more assurance of its success before marketing their products. Some advances have been made and are worth reporting at this time.

The role of bacteria

Basic studies have been conducted with films, foils and lamination combinations which indicate that flexible films may be permeable to bacteria. Proctor and Nickerson (3) have reported on the frequency of

Figure 1. Experimental flexible packages for heat-processed foods as tested by the U.S. Army Natick Laboratories. Surfaces of foil-laminate inner pouches are pulled back here to show contents.



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¹Numbers in parentheses identify References appended.

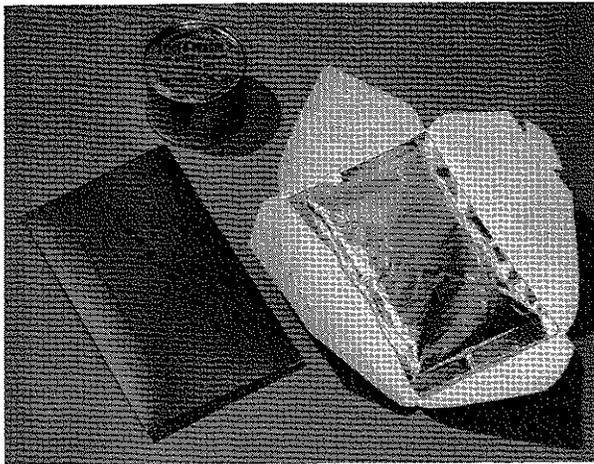


Figure 2. Heat-processed pouch in paperboard folder is being studied as replacement for metal can in field rations.

penetration of a variety of materials (plain, creased and heat sealed) when exposed to microorganisms ranging in sizes from 0.5 to 4 microns in diameter. The conclusions reached showed:

1. In general, plain films except those of small gauges (0.5 mil or less), showed no penetration by microorganisms.
2. Plain aluminum foils showed microbial penetration.
3. Creasing tends to increase the penetration, particularly with materials of low gauges.
4. The frequency of penetration was not increased by heat sealing.

The results of the tests led to the conclusion that penetration of films by microorganisms occurs because of flaws in the material.

Later, Hartman, et al. (4), showed that bacteria can pass through some plastics films. Although the contamination was generally low, the films were permeable to microorganisms. The basic question of whether bacterial passage was the result of undetectable pinholes or actual permeability, however, was unanswered.

These investigators suggested that factors other than pinholes are involved. Regardless of the mode of passage, bacteria did pass through some films and this is of great practical significance.

Experimental

Realizing the possible health-hazard implications, we conducted performance tests to determine the significance of microbial penetration with respect to laminations. The material used in the pouch was a laminate of 0.5-mil polyester/0.35-mil aluminum foil/3-mil vinyl.

Individual flexible packages were field tested under accelerated combat-use conditions and subsequently stored to determine whether microorganisms would

penetrate the packages (5). After six-months' storage there was no evidence of spoilage due to bacterial recontamination.

In addition, flexible packages as a component of a field ration were tested (6). The objectives of this study were to determine whether the packages could withstand the shipping, storage and environmental hazards expected in an overseas shipment and subsequently withstand microbial invasion.

After abuse and rough handling, the flexible packages were inspected for damage. Those that were not visible leakers were submerged in a tryptose-soyone media containing *E. Coli* and *A. Cloacae* for 48 hours. After exposure they were incubated at 37 deg. C. and examined every 24 hrs. for a 30-day period. Those exhibiting definite swelling were considered failures. Approximately 8% of the packages tested in this manner were failures. The microbial test indicated other factors than rough handling, such as questionable test procedures, and an appreciable influence on the performance of the packages. Controls of the test were samples that had been punctured with a 24-gauge hypodermic needle. From a total of 48, one-half showed evidence of swelling after test. Why didn't the other punctured packages show evidence of swelling?

It has been suggested that surface-tension effects create a barrier to the passage of bacteria when holes are very small and, further, heavier films possess self-sealing characteristics which resist entry. These could well be the answers, but supporting evidence is lacking. Based on the findings of this study, the tests were inconclusive. Sufficient data were not obtained to determine the complex nature of the microbial problem; however, it did reveal weaknesses which need further examination.

Recognizing the need for more basic information, we have entered into an agreement with the Continental Can Co. to study methods of determining bacterial contamination of foods processed in flexible packages. This study will be directed toward developing a test method which could be adopted as a standard in evaluating the integrity of a pouch. Its measured performance will be comparable with that of a can.

Extractives

The behavior of the packaging material when subjected to the conditions of processing from an extractive standpoint is another important consideration when discussing its integrity. Materials used in contact with food must satisfy Federal Regulations for safety. For our purpose, we have established that plastics-foil laminations are essential to meet the military requirements; therefore, to comply with the Federal Regulations, the materials must satisfy the requirements

specified in Section 121.2514, Resinous & Polymeric Coatings, of the Federal, Food, Drug & Cosmetic Act.

Migration or leaching of components from flexible packaging materials were investigated by Karel and Wogan (7). The foil-bearing materials tested to meet high-temperature processing conditions, using simulated food solvents such as water, acetic-acid solutions and n-heptane, showed:

1. Three polyolefin-foil materials were acceptable and three vinyl materials were unacceptable with regard to F&DA regulations.

2. Infrared analysis of heptane extractables from the polyolefin materials suggested the residues were long-chain hydrocarbons which had been partially oxidized; whereas from vinyl materials the residues appeared to be aryl esters, probably phthalate.

3. Solvents tend to remove more extractables from heat-sealed films and vary with the type of solvent.

Although heat sealing increased the amount of extractables, the total amounts extracted were below the minimum acceptable levels under Federal Regulations. The fact that this occurred points out that, if borderline cases were involved in the ordinary extraction of materials, increases due to heat sealing could produce amounts in excess of the acceptable levels. The results showed that extractive procedures using standard cells utilizing sheet material could be significantly different from the results if heat-sealed pouches themselves were employed.

In addition to standard solvent extractions of the materials, an attempt was made to determine the validity of using the five-to-one fixed ratio between the amount of heptane extractables at 150 deg. F. to oil at 250 deg. F. and oil at higher temperatures. As stated in the Regulations: "Heptane extractivity results must be divided by a factor of five in arriving at the extractivity for a food product."

Several analytical methods were tried in which oil extracts were compared with heptane extracts and oil to which heptane extracts were added. These were:

1. Ultraviolet analysis
2. Chromatographic analysis
 - a. Column chromatography
 - b. Thin-layers chromatography
3. Gravimetric-difference method
4. Gravimetric determination of non-saponifiables
5. Infrared analysis
6. Nephelometric analysis

By the latter method, the ratio of heptane extractables at 150 deg. F. to oil extractables at 250 deg. F. was found to be 3:2; at 275 deg. F. the ratio was 1:6; at 285 deg. F. it was 0:2, and at 300 deg. F. it was 0:5. It appeared from the results that the ratio of heptane extractables at 150 deg. F. to oil varies with the temperature of the oil.

From the above, it can be concluded that the factor



Figure 3. Presence of gas-producing organisms is evidenced by "swellers" such as these.

of five is not realistic for plastics films. Since this factor was derived for canned enamels, its applicability is dubious. As a result, several areas which warranted further investigation were:

1. To develop methods for the accurate determination of extractives from packaging materials in contact with oil.

2. To establish a meaningful ratio between the extractability for food products having water-in-oil emulsions, free oil or fat, to pure solvents when the condition of use is 250 deg. F. and higher.

3. To evaluate the significance of off-flavors and off-odors extracted from packaging materials.

Currently, we have a contract with The Pillsbury Co. to conduct further studies in these areas. Foil laminations with the following food contact surfaces will be tested: polycarbonate, polyamide, polyester and fluorocarbon.

Sealability

The effectiveness of a container is largely dependent upon the sealant that bonds it together. Since processed foods require the container to be hermetically sealed for protection against air, contamination and foreign materials, its effectiveness is usually measured by leakage. Sealability is, therefore, a major factor in establishing the integrity of a container and in the case of the flexible package must be the same as the can.

Early in our development of a flexible package for processed foods, we searched for a method to seal flexible barrier materials. Christie, et al. (8), investigated various methods of sealing: adhesive, heat and ultrasonics. A limited amount of work was expended in the evaluation of adhesives for package sealing. The performance of a urethane adhesive was very good; however, there were objections to it based on the toxic nature of the isocyanate component. The results of work were unrewarding and showed that the development of high-strength seals by adhesives is very difficult.

Heat sealing was more promising. Experiments

were conducted showing the factors that are important to produce optimum seal strength, such as: plastics type; plastics thickness; seal direction; sealing temperature, pressure and time. Highly efficient heat seals (95%) were obtained with polyester-foil-vinyl laminates.

The effects of accelerated storage on seal strength were studied. Indications were that polyethylene strength declined rapidly in storage, while polyvinyl chloride retained most of its strength.

The effects of food-particle contamination on seal strength were also studied. Analysis of the data showed that polyethylene is more susceptible to loss of strength than polyvinyl chloride. Furthermore, seal-strength loss varied with the type of contaminant. Solid contaminants tended to have a greater effect than liquid. There was even a significant difference between the action of water and oil, water having the greater effect.

The results in the application of ultrasonic energy to sealing were discouraging. Limitations on its use (films and foils, but not laminations), high cost and complexity of the technique were found objectionable. This is, however, a relatively new field and much research is currently being performed which could change the picture in its entirety.

We might conclude from the studies which have been made to date that:

1. There is no generally accepted standard for seal strength at this time.
2. The optimum obtainable with existing sealing equipment for a given material has been considered satisfactory.
3. To attempt to set a standard would be difficult and impractical with all the variables involved.
4. Therefore, to insure that you have an effective container, performance or field testing under actual use conditions is the most practical approach to this problem.

Field tests were conducted recently by the U. S. Army General Equipment Test Activity, Fort Lee, Va. (9, 10). Used were new and different products as well as new and different materials. Products included green beans, whole kernel corn, chicken a la king, fruit cake and date pudding. The pouch material was a lamination consisting of 0.5-mil polyester/0.35-mil aluminum foil/3-mil polyolefin. Evidence was secured that this flexible package was sufficiently durable to withstand simulated combat conditions. Leakage in the tests was less than 10% and considered satisfactory under the severe conditions of test. The packages were subjected to abuse equivalent to a full 10 days in the field, whereas only one day was required. For all products except green beans and whole kernel corn the abuses given did not affect product identity. Exposure to 10 traversals of the combat

course crushed the green beans and corn. This condition was partially attributed to the use of frozen products during initial packaging.

Conclusions

What criteria should we use to determine the safety of packages? Must one microscopically examine the contents of each package to establish its safety? Spoilage in the flexible package is no different from that in a can; therefore, the same criteria that are used for cans should be employed for the flexible package. If spoilage occurs, changes in appearance, odor, flavor and texture will become apparent (Figure 3). When gas-producing organisms exist, swellers will develop (11).

The integrity of a package is what we make it. In the case of heat-processed, flexible food pouches, continued studies pertaining to bacterial penetration, extractives and sealability are paramount.

References

1. Rubinate, F. J., 1964, "Army's 'Obstacle Course' Yields a New Look in Food Packaging," *Food Technology* 18 (11).
2. Robe, K., and Mayer, P. C., 1963, "Canning Without Cans." *Food Processing*, Nov., p. 75.
3. Proctor, B. E., and Nickerson, J. T. R., 1958, "Investigation of Bacterial Resistance of Packages," Report of QM Research Contract DA-19-129-QM-758, M.I.T., Cambridge, Mass.
4. Hartman, N. W., Powers, J. J., and Pratt, D. E., 1963, "Bacterial Permeability of Selected Food-Packaging Film," *Food Technology* 17 (9) 92.
5. Burt, T. B., 1963, "Engineering Test of Packaging, Flexible, for Heat-Processed Beefsteak," unpublished. U. S. Army Test & Evaluation Command, Fort Lee, Va.
6. Anonymous, 1961, "Performance Evaluation of Flexible-Packaged Rations," unpublished. QM Food & Container Institute for the Armed Forces.
7. Karel, M., and Wogan, C. N., 1963, "Migration of Substances from Flexible Containers for Heat-Processed Foods," Report of QM Research Contract DA-19-129-QM-2080, M.I.T., Cambridge, Mass.
8. Christie, H. W., Boleze, C. C., and Medved, T. M., 1959-1961, "Development of Methods for Sealing Flexible Containers," Report of QM Research Contract DA-19-129-QM-923, Midwest Research Institute, Kansas City, Mo.
9. Brugh, J. F., 1964, "Engineer Design Test of Flexible Packages for Heat-Processed Foods—Beans, Green; Corn, Whole Kernel; Chicken a la King," unpublished. U. S. Army Test & Evaluation Command.
10. Brugh, J. F., 1964, "Engineer Design Test of Flexible Packages for Heat-Processed Foods—Fruit Cake and Date Pudding," unpublished, U. S. Army Test & Evaluation Command.
11. Anonymous, 1963, "Spoilage Detection of Retort-Packaged Products—MID," unpublished, Continental Can Co.

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