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# Determination of Global Extractives in Polyethylene Terephthalate by Heating the Solvent in a Microwave Oven

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Rochester Institute of Technology

Department of Packaging Science

Determination of Global Extractives in Polyethylene Terephthalate

by Heating the Solvent in a Microwave Oven

by

Anuj Vinodchandra Parikh

A thesis, submitted to

The Faculty of Department of Packaging Science,

in partial fulfillment of the requirements for the degree of

Master of Science in Packaging Science

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Title of Thesis: The Determination of Global Extractives in Polyethylene Terephthalate by Heating the Solvent in a Microwave Oven

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Date: April 11, 1988

**Anuj V. Parikh** 

## **ABSTRACT**

# DETERMINATION OF GLOBAL EXTRACTIVES IN POLYETHYLENE TEREPHTHALATE BY HEATING THE SOLVENT IN A MICROWAVE OVEN

By

Anuj Vinodchandra Parikh

The purpose of this research is to evaluate a method of determining extractives. Instead of a conventional oven, a microwave oven is used to heat the solvent.

Attempts are made to duplicate the extraction methodology proposed by the Food and Drug Administration (FDA) in a way that it can be used to determine extractives for microwave-only packages. <sup>A</sup> test package consisting of crystalline polyethylene terephthalate and paper was selected for the test. A product consisting of a dehydrated mix of pasta and cheese sauce was evaluated to determine the test parameters including the solvent, time, and temperature.

Distilled deionized water was selected as a solvent. It was exposed in two different package shapes to counter any microwave heating irregularities. The solvent was heated in packages for 10 minutes in a 700 watt microwave oven at maximum power. The extractives, in each case, were obtained by evaporating the solution under reduced temperature of  $60^{\circ}F$  (15.5 $^{\circ}C$ ) and in vacuum, a method commonly referred to as vacuo. <sup>A</sup> Rotavapor machine was used for this purpose. The extractives were then dried and weighed. They were further dissolved in chloroform. Chloroform-insoluble extractives were filtered out and further extraction was performed on the solution. The temperature was maintained at  $35^{\circ}F$  (1.7 $^{\circ}C$ ).

It was found that the amount of extractives obtained by the conventional FDA method were comparable to the ones obtained by this procedure. In both cases the amount of total extractives for this package fell within the specified limits set by the FDA in milligrams per square inch of contact surface.

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# DETERMINATION OF GLOBAL EXTRACTIVES IN POLYETHYLENE TEREPHTHALATE BY HEATING THE SOLVENT IN A MICROWAVE OVEN

#### **INTRODUCTION**

Extractives are those compounds that migrate from a plastic material when it is subject to external energy. In food packaging applications this energy comes in the form of heat, normally generated by ovens. The Food and Drug Administration (FDA) has established guidelines to limit such migration and has outlined tests to regulate the quantity of migrants that come in contact with food at elevated temperatures.

In microwave ovens, generation of heat and, consequently, the cooking of food is based on the conversion of electromagnetic energy into heat energy within the product. Unlike conventional ovens where heat is imparted by way of conduction and convection, microwave ovens project electromagnetic waves to the product whereupon they agitate the molecules. This results in friction and generation of energy in form of heat.

According to J. Steiner (1982), today there is a tendency to make food packages microwave compatible. Attempts have also been made to develop 'microwave-only' packages which are not suited for use in conventional

ovens. Microwave-only packages are more economical in terms of packaging material used. As a result the package is economical but efficient enough to serve its purpose of containing and protecting the food while it is being cooked, and during packing and distribution.

A number of polymers are deemed suitable for the purpose of microwave cooking where the polymer comes in direct contact with food. These include polyesters like polyethylene terephthalate (PET) and polybutylene terephthalate (PBT), high impact polystyrene (HIPS), polypropylene (PP), polyethylene (PE) and nylon. The FDA has cleared some grades of these materials for use in contact with food at elevated temperatures. In order to obtain commercial clearance, users of packages made of these materials have to submit extraction data to the FDA. The tests devised by the FDA for polyethylene terephthalate include subjecting the packaging material and an appropriate solvent to 250 $\mathrm{P}F$  (121 $\mathrm{C}C$ ) for two hours. For a package that is designed to be subjected to microwave environment only, and that too for a significantly short period of time, it is a stringent test and prevents package manufacturers from investigating packages in many different configurations of thickness and coatings.

The specific tests outlined by this research are to serve as a guideline for the FDA which according to T. Begeley (1987) is in a process of outlining an

extraction test for microwave-only packages. This test is devised for a particular product package system and may not be appropriate to test other materials or other products.

The packaging material selected for food contact surface in this test was crystalline polyethylene terephthalate (CPET) with approximately 12% crystallinity. The package was thermoformed of 0.95 intrinsic viscosity resins. Two distinct shapes were selected to consider all possible microwave cooking characteristics. The packages selected were a square package with dimensions 3-3/4" x 3-3/4" x 2-5/8" (96.2mm x 96.2mm x 67mm), and a rectangular package with dimensions 5-11/16" x 3-3/4" x 1-3/8" (145.5mm <sup>x</sup> 95.5mm x 35mm).

The low crystallinity of the polyester results in cost saving in terms of energy consumption in the manufacture of the base sheet material. For a dual ovenable package, crystallinity in the range of 25-35% is required to withstand a cooking temperature of above  $400^{\circ}F$  (204 $^{\circ}C$ ). The test package is given structural support by a paper layer laminated to it by use of a hot melt adhesive during the thermoforming process. With five paper sides acceptable to printing, the package is suitable for microwave-to-table use and can be used for low - medium priced menu items that require microwaving.

As specified in a study by H. Rubbright (1986), CPET has emerged as a very effective material for microwave-only packaging, particularly in today's market where special food products are being developed to suit microwave oven cooking. The significance of CPET as a microwaveable material was also noted by R. Schiffmann (1982).

Apart from food packaging, microwaves were used in many applications for polymers. Examples have been found where microwave drying of polymers was studied by N. Vasilakos and E. Magalhaes (1984). Microwaves have also been utilized for drying of coated films by E. Stephensen (1972) and for low temperature processing of plastics by G. Lighesey, C. George and L. Russell (1986). With its inherent properties CPET was the logical choice for high temperature cooking applications.

#### **Objective**

This thesis will specify and demonstrate the use of a new testing method to determine the total extractives obtained by microwave heating of a solvent in contact with thermoformed crystalline polyethylene terephthalate material. The solvent will simulate a dehydrated pasta and cheese product. The objective of the research is to compare the results obtained by conventional heating of the solvent against microwave heating, and determine whether the global extractives obtained by this methodology fall within the limits specified by the FDA.

#### LITERATURE REVIEW

In a recent survey conducted by M. Kass (1986) in Packaging magazine, it was found that 65.3% of the respondents use a microwave on a daily basis. The microwave is being used for reheating leftovers, cooking, baking, and heating liquids. Also found was the fact that the respondents tend to use plastic and paperboard trays to cook food in a microwave, more than any other material.

As J. Briston (1986) mentioned, package manufacturers have come up with many new configurations to combine the advantages offered by paper and plastic in packages that are both easy to use and economical. The food industry has been quick to welcome this change as the perception of paper/plastic packages changes for the better. According to an article, "LaChoy has switched entirely from foil to ovenable paperboard trays for its Chinese dinners and entrees" in Prepared Foods (1982), conversion from foil to plastic or paper composite package is rapid, and as the materials become more economical the demand is bound to rise. Techniques such as extrude-into-mold thermoforming, as described in "Extrude-into-mold process makes better containers" in Packaging Digest (1986), co-extrusion for aseptics and other applications outlined by B. Miller (1983), lamination for barrier, and resin technology are producing better, cheaper packages that

can be made to exact specifications as shown in "CPET food trays see more action" in Packaging (1985). These packages can be dual ovenable or for microwave use only. The lack of excess material makes the microwave-only package more economical in terms of energy consumption during forming and actual oven use. The saving in material alone is also significant as pointed out by "Thermoformed plastic-board pack is bonded" in Packaging Review (1969). This has resulted in a boom in microwave cooked foods. J. Rice (1987) has found that entree preparation in microwave ovens has increased 90% over the past 3 years and Stouffers, Campbell, Pillsbury, General Foods, and other food processors have a number of packaged products designed for microwave use only.

Pasta is a complete food that is extremely versatile when it comes to methods of preparation and serving. An industry report by B. Messenger (1987) states that a number of food processors are actively pursuing the microwave cooking of pasta entrees. <sup>A</sup> statistical survey by N. McCue (1987) has revealed that today, the pasta business is a <sup>1</sup> .3 billion dollar market that should grow to 1.6 billion dollars by 1990, or from 2.6 billion pounds in 1985 to 3.3 billion pounds expected to be sold per year by 1990. The annual per capita consumption of pasta in the U.S. is <sup>1</sup> 0 pounds today. This would include any of the 600 shapes or sizes of pasta currently available. At least 77% of the total volume is packaged in one form or another.

B. Messenger (1987) in a survey conducted for Prepared Foods found that today well over 50% of households own a microwave and 87.5% of consumers who own microwaves look first for microwaveable products. This has prompted introduction of 288 new microwaveable products in 1986. In 1987, through July only, 439 microwaveable products were introduced which accounted for over 10% of the 4366 new products introduced during that period. In numerous cases food companies have tried to take advantage of this microwave boom by only changing the package to being a microwave compatible one and lableling the product as 'microwaveable'.

It is clear that there is an acute need for testing microwaveable packages in their environment of use, that is, the microwave itself. This thesis will attempt to do so by determining the total extractives that may migrate from a polyester, CPET, material to a product consisting of a dehydrated mix of pasta and cheese sauce, to be cooked in a microwave. The package selected for this purpose is Tritello, manufacutured originally by Akerlund and Rausing in Sweden, and in the U.S. by Sonoco Products Co. The package is made up of a preprinted paper blank, which is heat sealed to a thermoformed plastic liner. As mentioned in "Sonoco will develop U.S. market for Tritello' paper/plastic container" in Packaging Strategies (1987), though polyvinyl chloride (PVC) was generally used in Europe to package dairy products, Sonoco is experimenting with CPET, HIPS, PP, polyethylene terephthalate

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glycollate (PETG), and other barrier materials. For all experimental purposes in this paper, CPET shall be considered as the material selected for testing.

Apart from being available in a wide variety of sizes, a survey conducted by Elrich and Levidge in 1985 found that the package is appealing to most consumers as a convenient, easy to use package that can be used for entrees as an oven-to-table package. It is most suited for solid or dry food products.

The testing will focus on determining the adaptability of the package to microwave environment. The basis of judgment will be the amount of extractives obtained in a microwave as compared with those obtained by the conventional FDA methodology.

#### 2.1 Migration

Migration can be considered as a mass transport process under defined test conditions which include time, temperature, and nature and volume of contacting phase. It is a complex process depending on diffusivity (D), which is defined as, 'the tendency of the substance to diffuse through the polymer bulk phase'. According to J. Giacin and J. Miltz (1983) diffusivity depends on the concentration gradient where the dissolved material diffuses from a high concentration region to one with lower concentration due to the action of

equilibrium forces. Thus for migration to occur, a particular chemical would have to diffuse through the polymer to the contact phase and dissolve or evaporate into the contact phase.

Extraction of packaging material as specified in ASTM F-34 "Standard test method for liquid extraction of flexible barrier materials" or extraction of formed packages outlined in FDA, Code of Federal Regulations (CFR) <sup>21</sup> , 177.1630, FDA Ch.1 "polyethylene pthalate polymers" remains one of the most important test characteristics for plastics used to package foods, particularly when they come in contact with foods at elevated temperatures during cooking.

The detection of such migration can be carried out in two distinct ways, one being the determination of global migration. Global migration is the total transfer, in milligrams, of all substances migrating from the package into the solvent or the contact phase. The second method is the determination of specific migration. It is the detection or analysis of one or more identifiable chemicals through qualitative extraction. It can be done in a variety of methods, the most common being the injection of solvent or the extractive into the gas chromatograph and obtaining the results through mass spectroscopy. Migration can be attributed to the transfer to base materials or trace constituents. Some possible migrants are listed in table 2-1 .

## Table 2-1 . Possible migrants from the packaging material into the product.

Residual monomer Low molecular weight polymer **Catalyst Antioxidants** Colorants Residual solvents **Defoamers** Light stabilizers Reaction products

(Modern plastics encyclopedia, 1983)

Lubricants and slip agents Antiblock agents **Plasticizers** Antistatic agents Blowing agents **Emulsifiers** Chain transfer agents Polymerization inhibitors Decomposition products

In this study the concern is with global migration only. It is the gravimetric determination of extractives obtained by placing the package and solvent under defined time-temperature conditions.

<sup>A</sup> number of regulating bodies have published parameters for determination of global migrants through extraction. The methodology varies with the type of material and product to be tested. Administrators have tried to simulate the fiercest conditions that the package would face during transport or storage and based their tests accordingly. The tests for packages that can be used for cooking food in a conventional oven or a microwave are more exhausting. Some of the agencies and their regulations are cited in the European Legislation Update-Food Packaging and Labeling by S. Sacharow (1987), and Migration from Food Contact Plastics, Part I. Establishment and aims of the PIRA project by P. Tice and J. McGuinness (1982).

The Food and Drug Administration has defined "food additive' as "any substance, the intended use of which results or may reasonably be expected to result, directly or indirectly in its becoming a component or otherwise affecting the characteristics of any food". According to this interpretation, extractives are 'food additives' and limits have been established by the FDA for maximum allowable extractives in milligrams per square inch of contact surface. In this study the concern will be with the extraction from CPET

material. If there is an interest for the determination and analysis of extractives of other materials such as polystyrene, acrylonitrile, and PVC, refer to "Analytical Measurements of Package Components for Unintentional Migrants" by J. Giacin and A. Brzozowska (1985).

#### 2.2 Analysis of Extractives

The commercial production of polyethylene terephthalate from terephthalic acid and ethylene glycol involves two steps. The first step consists of an esterification reaction between terephthalic acid and ethylene glycol in presence of a catalyst to form a prepolymeric mixture which then undergoes polycondensation in the second step. The esterification product prepared in first step contains bis-(2-hydroxyethyl) terephthalate (BHET), ethylene glycol (EG), diethylene glycol (DEG), terephthalic acid (TA), mono-(2-hydroxyethyl) terephthalate (MHET) and higher molecular weight oligomeric polyester. The reaction involves no external reagents, catalysts or solvents, thus there is no possibility of such migrants. E. Atkinson, Jr. and S. Calouche (1971) have proposed a qualitative and quantitative procedure for the determination of MHET, BHET, EG, DEG, and TA in polyester prepolymers by conversion to their trimethylsilylation (TMS) derivatives followed by gas liquid chromatography (GLC).

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Louis M. Zabrosky II (1977) used high performance liquid chromatography (HPLC) for the determination of PET prepolymer oligomers containing from one to seven terephthaloyl repeat units. According to T. Begeley (1987) the compounds with dimer and trimer are the most common ones to occur. S. Jabarin and D. Balduff (1982) described a gel permeation chromatography (GPC) technique for the measurements of cyclic trimer content, molecular weights and molecular weight distribution of PET. Thus in order to determine all possible monomers and oligomers present in a PET extract, C. Nelson (1987) specified two distinct methods. <sup>A</sup> gas chromatography (GC) procedure to identify EG, TA, DEG, BHET, and MHET and HPLC procedure to determine high molecular weight oligomers of HET.

#### MATERIALS AND METHODOLOGY

#### 3.1 THE PACKAGE

As described in the "Fact Sheet-Tritello" published by Sonoco Products Co., the test package consists of a thermoformed plastic liner on the inside, heatsealed to a preprinted paper surface on the outside. The plastic (CPET) acts as a food contact layer while the paperboard provides rigidity and five fully printable surfaces.

Two configurations are selected to study the effects of microwaves on square and rectangular packages respectively. This provides a way to study the differences in heating rates in relation with the surface area of the solvent. The capacity of the square package is 376.9 ml with an overflow capacity of 410 ml. The rectangular package has a capacity of 357.3 ml with overflow capacity of 420 ml. This is to accomodate 76.5 grams of product and <sup>1</sup> 70 ml of water.

The package is made by thermoforming a CPET sheet into a paperboard tray. A hot melt adhesive is used to bind the layers together. CPET was selected as the material of choice due to its versatility in retaining its strength and structure at very low to very high temperatures. PET is approved for food contact by the FDA and is not known to scalp or impart any taste or odor to

the product. It is seen as the most stable material for microwave boiling of water and rapid, even cooking of the product.

The package is formed of 20 mil (0.020 inch) PET sheet. The paperboard layer gives the package its rigidity. In this respect a <sup>10</sup> point (0.010 inch) paper was considered adequate and is used.

#### 3.1.1 Raw Materials

One of the resin types that can be used to manufacture the test package is Goodyear's Traytuf 9506C PET resin. Refer to table 3-1 . for properties of this resin. It is a high molecular weight PET resin optimized for high strength and impact resistance. According to the manufacturer, the advantage of a polyester resin is that it can be crystallized within normal thermoforming temperature range and it retains its crystallinity until the polymer melts at approximately 480°F.

CPET is produced by melt phase polymerization of terephthalic acid and ethylene glycol, followed by a secondary polymerization process. It conforms to FDA regulation 177.1630 "polyethylene pthalate polymers" for food contact applications. The resins readily transmit microwave radiation and they are odorless as specified by J. Carty in his letter (1987).

# Table 3-1. Properties of PET resin (Goodyear Traytuf 9506C).

Intrinsic viscosity 0.95<br>
Melting point 257% Color, Gardner b 6.0 (maximum)<br>Aldehyde, ppm 2 (maximum) Aldehyde, ppm<br>Particle size

 $257^{\circ}$ C (minimum)<br>6.0 (maximum) Particle size **Approximately 1/8"** cubes The determining factors in PET for its physical properties and uses are its molecular weight and molecular weight distribution. As seen from the Jabarin and Balduff study (1982), the most frequently used technique in the industry to evaluate molecular weight of PET is through viscosity. The intrinsic viscosity or the limiting viscosity number, {n} is related to molecular weight, M, by Mark Houwink equation

$$
\{n\} = KM^{-a},
$$

where <sup>K</sup> and a are empirical constants.

The higher the stated I.V., the higher is the molecular weight of the resin. In conversation with J. DeGross (1987) it was found that loss of I.V. in the resin leads to rapid crystallization and subsequent loss in toughness of the material resulting in bad performance at low temperatures. Higher I.V. leads to slower crystallization resulting in better performance. Slower crystallization can also be obtained by leaving the material in the mold for a longer period of time. Temperature in the mold can be lowered by as much as 5 to 10  $\textdegree$  F to even out the energy cost. The material is tougher in this case. For experimental purpose, resin with 0.95 I.V. was used.

The 10 point SBS paper is a high quality paper manufactured by kraft process on Fourdrinier machines. The machine is particularly applicable to hardwood which has a much shorter and finer fibre than pine. <sup>A</sup> blend of 80% hardwood and 20% pine yields a balance in strength and finished

smoothness of the paper. This blend sacrifices very little in strength but obtains desired smoothness and thus is able to take advantage of other attributes of paper such as scorability and high quality graphic printing. The carton board is then die cut in desired forms.

#### 3.1.2 Thermoforming:

The resin and nucleating agent are blended in 97:3 ratio at the throat of extruder. Blended resin is then extruded into a sheet of desired thickness. In order to develop crystallization in the material, a heated mold is used for thermoforming. It controls the rate and degree of crystallization, producing heat stable crystalline containers. For test package, the sheeting is heated to approximately 300 $\degree$ F and mold temperature of 250 $\degree$ F is utilized to obtain about 12% crystallinity. The exact rate and degree of crystallization depends on time in the mold. The CPET tray is thermoformed into a die cut paper sheet that has hot melt adhesive applied to it. Upon heat activation, the adhesive sticks to the polyester and forms a heat stable bond. This produces a package that exibits adaptability to a variety of products that can be hot or cold filled, or gas flushed. Shelf life and permeability can be regulated by using a high barrier material or using CPET of higher thickness. Also pigmented plastics can be used to impart color to the package. The package is printable on five sides and it is nestable for easy storage of empties in the warehouse.

#### 3.2 PRODUCT: PASTA AND CHEESE

The solvent used for extraction will simulate a specific product. The product selected consists of a dehydrated mix of pasta and cheese sauce. As seen from the data given in table 3-2, the product is low in fat and high in carbohydrates. The pH of the prepared product is 5.8. The fat content of the product is less than 8%.

Recommended cooking directions for this product include adding 170 grams (3/4 cup) of water to 76.5 grams of product and heating it in a microwave for 4 to 5 minutes at high power, stopping to stir once after 2 to 2 1/2 minutes; or heating it in a microwave for 6 minutes at medium, stopping to stir twice, after 2 and 4 minutes.

In order to determine a single method of cooking the product on which to base the extraction methodology, both square and rectangular packages were tested according to the above specified cooking directions. The second procedure was found acceptable, whereupon the product was cooked evenly and after one minute of standing time it was cool enough to eat. The texture of the product was acceptable. When cooked at high for 5 to 6 minutes, the product was found to overheat and tended to bubble over and out of the package. Materials used for this test were the test packages, the

product, Kenmore 600 Watt microwave oven and distilled water.

# Table 3-2. Composition of pasta and cheese product.

Product: Cheddar Cheese Sauce and Noodles



#### 3.3 MICROWAVING

In a microwave oven the product is placed in an electromagnetic field and the heat energy is generated by the product's own ingredients. As reported by M. Perry (1987), this is because the molecules that carry a dipolar electrical charge, such as water, vibrate as they attempt to align themselves to a rapidly fluctuating electromagnetic field. The frequency of the electric and magnetic waves oscillating perpendicular to each other, is 12.24 cms in air. That is, the fluctuations of the field go from 0 to <sup>1</sup> approximately 2.45 billion times each second or with a frequency of 2450 megahertz. Consequently, according to a report by D. Best (1987), ingredients exibiting little electric polarity such as fats, will display poor dielectric lossiness and thus will generate less heat in the product. Also, charged ions such as H+ and OH- in water molecules or Na<sup>+</sup> and Cl<sup>-</sup> in salt will flux towards the rapidly alternating poles of the electric field contributing to the heat of friction as shown in figure 3.1 (A) and 3.1 (B). Thus different foods heat up at different rates depending upon their adsorption characteristics. This is shown by table 3-3 giving relative values.

#### 3.3.1. Microwave-Components:

In 1972, R. Decareau described various components of a microwave oven and their functions. All microwave ovens basically consist of a cavity, the door, the magnatron, the waveguide, the mode stirrer, the power supply and the power chord. The power supply converts the low voltage line power to



Figure 3.1 (A) Directions of magnetic and electric fields



Figure 3. <sup>1</sup> (B) Dispersion and allignment of ions under microwave radiation

# Table 3-3. Microwave absorption of food components.

Salt water (8%) 1.3<br>Salt water (2%) 0.9 Salt water (2%)<br>
Meat. fish and vegetables<br>  $0.25$  to 0.35 Meat, fish and vegetables 0.25 to 0.35<br>Distilled water 0.12 to 0.15 Distilled water 0.12<br>Oils and fats 0.06 Oils and fats

Microwave absorption<br>1.3

(R. Goddard, 1986)

the high voltage required by the microwave energy generator, the magnatron, which in turn generates high frequency energy that passes down the wave guide and enters the cavity. The mode stirrer interrupts the energy waves as it enters the cavity and causes it to be distributed more uniformly before it is absorbed by food. The food load is positioned slightly off the floor. This causes some of the energy to be reflected from the oven floor into the food from the bottom resulting in even cooking.

M. Perry (1 986) has stated that the temperature that a localized area of a food product, within a a package can attain is a balance of many factors including the energy input by the microwave's electrical (E) and magnetic (H) fields; the temperature of the product and surrounding materials; the thermal conductivity of the product and its contact materials; surface area of the product; surrounding air temperature; convection to surrounding air, and evaporative cooking. The temperature is also dependent on the shape of the package and the surface area exposed to the microwave radiation.

Some of these factors will vary with microwave type, watts and volume, but it is safe to assume that for a defined product/package system, the highest temperature will be reached in a microwave with highest output in watts.

#### 3.3.2. Microwave-Selection

The purpose of microwave testing is to determine how well the product cooks in any particular microwave oven. If the product is to be cooked in the microwaveable package itself, the testing becomes even more critical and care should be taken to be as accurate, precise and analytical as possible.

R. Schiffmann (1987) suggests that microwave ovens vary in, among other things, pulse time, power settings and turntables. In fact there is no single clear cut correlation between any single oven characterisitic and its cooking performance. Generally microwave ovens fall within the range of variables shown in table 3-4.

It would be impossible to analyze all the factors described in table 3-4 and include them in testing. The first two, namely the power output and cavity size are the most critical and will be analyzed. The microwave oven that generates the highest temperature in the product will be used to heat the solvent for extraction testing.

The three microwave ovens selected for testing were (1) a standard Kenmore 600 watt oven with <sup>1</sup> .2 cubic feet cavity size, it was a full sized oven without glass shelf, metal rack or turntable; (2) a Panasonic high power oven with 700 watts and <sup>1</sup> .6 cubic feet cavity size with a rotating waveguide and a

# Table 3-4. Variables in a microwave oven



turntable, the rotating waveguide provides an intense microwave field directed down at the product and that along with its high power output can cause stressful situations; and (3) an Amana subcompact oven with 450 watts and 0.5 cubic feet of cavity size without a turntable or a waveguide.

R. Schiffmann (1987) in his study mentioned that by using three such microwave ovens it would be possible to encompass a wide variety of features or performance factors that can influence the product/package performance. It can also simulate extremes in cooking cycles, power output and microwave field density. The same topic is discussed by A. Dungan (1987) in her study.

Two distinct tests were performed to evaluate the capacity of the microwaves. The first was devised by the International Electrotechnical Committee to determine the power output of a microwave. All three microwaves were tested in accordance with this method. One litre of distilled deionised water was placed in a glass beaker in the center of each oven and heated for 60 seconds at the highest power. The rise in temperature was measured and the power output was calculated by using the following formula,

P = 38.8 <sup>x</sup> At equation (3.2)

where P= power in watts and  $\Delta t$ = temperature rise in °F.

Initial water temperature was 70°F. An Omega thermocouple thermometer was used.

Results show that for microwave <sup>1</sup> , with 600 watts, the temperature of water after 1 minute was  $86^{\circ}F$ , a rise of 16 $^{\circ}F$ . Thus, according to equation 3.2, the net power output will be  $P = 38.8 \times 16 = 620.8$  watts. Similarly for microwave 2, 700 watts is the listed output. The temperature after <sup>1</sup> minute at high power was 89°F, a rise of 19°F, hence the net calculated output is 38.8 x 19 = 737.2 watts. After <sup>1</sup> minute of heating at high power, microwave 3 with 450 watts of listed power output showed a rise in temperature of  $14^{\circ}$ F up to  $84^{\circ}$ F. Thus the net power output is  $38.8 \times 14 = 543.2$  watts. Microwave 2 has the highest power output and should be selected for heating the solvent for microwave testing.

In the second method the product was cooked in the test package as per the instructions. Both square and rectangular packages were tested in all 3 microwaves. The product consisting of 40.6 grams of pasta and 35.9 grams of cheese sauce, was cooked with <sup>1</sup> 70 grams of water per package. After cooking, temperature was taken at <sup>10</sup> different points in each package with a thermometer having a digital readout for quick and accurate measurements. The results are listed in table 3-5.

## Table 3-5. Temperature at various points of the product cooked in the test package in a microwave oven.



In the 700 watt microwave, the highest temperature reached was 208°F. It was the highest temperature reached among all three microwave ovens tested. The product was more evenly cooked due to the presence of a turntable.

It is concluded from these two tests that microwave 2 has highest power output and generates highest temperature in the product. Thus it was the microwave oven used for heating the solvent for extraction testing.

#### 3.4 MIGRATION

Migration, in association with food and plastics, is defined as "the transfer of moieties from the package to the contained product." Factors affecting migration shall be studied later in this chapter. J. Giacin and A. Brzozwska (1985) have pointed out the distinction made between global migration and specific migration. Global migration involves the quantitative measurement of migrating substances, usually in weight units, whereas specific migration refers to identifying, qualitatively, one or more of the migrating chemicals that is a constituent of the packaging material. It is usually followed by analysis as to how that substance affects the quality of the packaged product. In this thesis the causes and detection method for global migrants will be studied; that is, the determination of transfer of all the substance, toxic or non-toxic, to the food contact phase, without analyzing them any further.

According to J. Miltz (1982), there are two basic approaches to migration. The older approach was based on the linear model assuming that whenever there is a residue in the package, it will migrate into the contents irrespective of its concentration. Several years ago a non-linear model was proposed where the residue in the polymer is composed of three different entities. First are the immobilized molecules. They are in low concentration and are tightly bound to the most active sites in the polymer and are therefore, non diffusable. Second are the partially bonded molecules. They are also bound to active sites but with much lower bond strength and thus, can migrate only under appropriate conditions, and third are the non bound molecules, which can easily diffuse through the polymer. In this model migration can occur only if the concentration of the residual spieces is above a certain threshold level.

T. Downes (1982) further divided the migrants into three classes based on their limiting control mechanism or diffusivity. Class <sup>1</sup> include the non migrating materials. They do not migrate irrespective of the presence of food. Diffusion coefficient is less than 10<sup>-12</sup>cm.sec<sup>-1</sup> at all concentrations of food. Class 2 consists of independently migrating materials. Presence of food may accelerate the migration of these materials, but it is not solely dependent on the food. Diffusion coefficient is between 10<sup>-9</sup>cm.sec<sup>-1</sup> to 10<sup>-12</sup>cm.sec<sup>-1</sup>. Class 3 are the leaching materials. Migration, in this case, is totally controlled by the presence of food at the contact surface. There is negligable migration in absence of food. Diffusion coefficient is less than  $10^{-12}$ cm.sec<sup>-1</sup> in absence of food but 10<sup>-9</sup>cm.sec<sup>-1</sup> or more in presence of food.

Broadly, migrants have been classified into two basic categories by J. Koszinowski and O. Piringer (1986). First is the migration of volatile organic materials from polymers. Along with a few other additives, this group consists of non-reacted monomers from the polymerization process. Examples include vinyl chloride from PVC, acrylonitrile from polyacrylonitrile, and styrene from polystyrene. In case of PET, they may be EG, TA, DEG, MHET, BHET and cyclic dimer or trimer compounds. The second category includes the migration and permeation of substances which are difficult to volatilize. These are not easily identifiable but in most cases include adhesives, coatings, inks and other surface contaminants that can migrate through absorption/desorption. Migration and permeation of these compounds depend on their chemistry and structure of the polymer.

## 3.4.1. Extraction: Establishing Parameters

According to R. Schiffmann (1987), "normal tests" are those that follow manufacturers instructions as they are written. Though in most cases this is true, some consumers may misinterpret instructions either accidently or through negligence. Abusive tests are designed to anticipate unusual

conditions which may arise and thus give data on what may occur when the product is handled abusively.

S. Sacharow (1987) and P. Tice and J. McGuinness (1987) have mentioned that the FDA, the European Economic Community (EEC), and other regulating bodies control and monitor the global migration through extraction. They tend to incorporate factors such as extra heating times, higher temperatures and strict limits on total extractives on their tests to simulate abusive handling of the product. This test shall also include means of higher temperatures and longer heating times in the microwave oven to incorporate abusive testing.

Selection of the package and the microwave have already been finalized in the previous sections of this study. Next, the appropriate solvent will be chosen to simulate the product in accordance with FDA guidelines and decision will be made on the time and temperature to which the package should be exposed in the microwave oven. Finally, the equipment to be used for extraction shall be reviewed. Selection of the solvent depends on the pH of the product and the fat content of the product. FDA has specified the solvents to simulate various products as shown in table 3-6.

# Table 3-6. Solvents used to simulate different products as specified bv the FDA.

Solvent **Product** 

Distilled water 3% Acetic Acid 8 or 50% ethanol n-heptane

Non acid foods, pH above 5.0 Foods with pH 5.0 or below Foods containing alcohol Fatty foods

(FDA guidelines for chemistry and technology, 1976)

The product, a dehydrated mix of pasta and cheese sauce has a pH of 5.8 after cooking and 6.8 before being cooked. The fat content of the product, including pasta, is less than 10% (high carbohydrate diet). Thus the solvent of choice will be distilled water.

According to FDA 21 CFR Ch.1 177.1630 (4-1-85 ed.), " polyethylene pthalate polymers" for plastics, such as PET, when the plastic is to be used to contain foods during oven baking or oven cooking at temperatures above  $250^{\circ}$ F, the food contact surface should be exposed to distilled water at 250 $^{\circ}$ F for 2 hours. In this test, 250 ml of distilled deionized water was subjected to different microwave exposure times . This was to determine the optimum time and temperature conditions for the actual heating of the solvent for extraction testing.

For this test a Panasonic microwave oven with 700 watt output was used at maximum power. Temperatures were taken within five seconds of the end of microwave cycle by a digital thermocouple thermometer. The cold water temperature was 75°F. Results are listed in table 3-7.



# Table 3-7. Comparative volumes and temperatures at different times in a<br>microwave.

As a result of this study 10 minutes of microwaving at high power was selected. At longer times a considerable amount of water is evaporated, and deterioration in the product is observed. Highest temperature observed during the actual cooking of the product is 208F, thus there is no need to go to longer times. At 6 and 8 minute marks, the temperatures required, of 208°F and above are not reached consistently.

Despite instructions, in some cases consumers tend to use high power instead of medium. To include this variable, all further testing including heating of the solvent for extraction will be done at high power.

The Rotavapor RE 120 by Buchi/Brinkmann was selected as the extracting equipment. Though a number of methods and equipment are suited to obtain the extractives from the solution, Rotavapor was selected for its ability to evaporate the solution at considerably below its boiling point. For example, water was evaporated at 60°F and chloroform at 35°F. This not only results in a very efficient and safe extraction but the process also prevents the highly volatile extractives from boiling off.

The equipment consists of a hot water bath into which the flask containing the solution is immersed. At the mouth of the flask is a rotator connected to the vacuum and a cold water bath. The solvent is vaporized at a temperature

lower than its boiling point by the action of vacuum and the rotator. The solvent is then separated into another flask by cooling it by means of a spiral cold water bath. Ultimately all of the solvent is separated into the other flask and only the extractives remain.

#### 3.4.2. Extraction-Testing

Detection of global migrants by means of extraction will be carried out in accordance with the FDA guidelines. The microwave extractives will be compared to the ones obtained conventionally. As specified in FDA 21 CFR Ch.1 177.1630, "polyethylene pthalate polymers" for polyethylene terphthalate material used to contain foods during oven cooking or oven baking at temperatures above 250°F, the food contact surface should be exposed to distilled water for 2 hours at 250°F. The limit for chloroform soluble extractives is 0.02 milligrams per square inch of food contact surface exposed to the solvent. The method of obtaining chloroform soluble extractives is specified in FDA 21 CFR Ch.1 176.170.

To obtain the total extractives by heating the solvent in a conventional oven, 250 ml of freshly deionized distilled water was heated in a gas oven for <sup>2</sup> hours at 250°F. The oven was preheated for ten minutes. Three round bottom flasks (no.1 -500ml capacity, nos. 2,3-1 00ml capacity), Rotavapor,

thermometer with digital readout, reagent grade chloroform, Whatman No. 41 filter paper, and Mettler $/E$  120 weighing scale were the equipment used.

The respective contact surface to the solvent for the square test package is 29.0 square inches and that for the rectangular test package is 36.2 square inches. The test packages of both shapes had CPET as a solvent contact layer . The material had 12% crystallinity and was formed of 0.95 I.V. resins. FDA <sup>21</sup> CFR Ch.1, 177.1630 and 176.170 were considered as a reference. Distilled deionized water was heated in the oven for 2 hours at 250°F in the two specified test package shapes. At the end of the exposure period, the packages were carefully removed from the oven and the solution emptied into a round bottom flask no. 1. The package was washed 3 times with distilled deionized water and the residue transferred to the flask no. <sup>1</sup> . The flask was attached to the rotavapor and the contents allowed to evaporate at  $60^{\circ}$ F. With about 90 ml of the contents remaining, the solution was again transferred into a tared flask no. 2. The bigger flask was washed and the residue transferred to the smaller one, which in turn was attached to the Rotavapor and further evaporation carried out. The last few drops were evaporated in the oven at a temperature of approximately 221°F. The tared flask no. 2 was cooled in a dessicator for 30 minutes and the extractives were weighed. Calculations were carried out to determine the milligrams of extractives per square inch of contact surface.

To obtain chloroform soluble extractives, about 50 ml of distilled reagent grade chloroform was added to the dried extractives, the solution was gently warmed and stirred in a round bottom flask no. 2. The contents were then filtered into a tared round bottom flask, no. 3, through Whatman No.41 filter paper. This seperates the chloroform soluble extractives from the rest. This organic solution was evaporated through vacuum at 35°F in the Rotavapor. The contents were nitrogen flushed to remove all possible free chloroform. The last few drops were dried in an oven maintained at  $221^{\circ}$ F. Flask no.3 was cooled in a dessicator for 30 minutes and the chloroform soluble extractives were weighed. Calculation was carried out to determine the milligrams of extractives per square inch of contact surface, a unit commonly used by the FDA.

To obtain total extractives through microwave oven heating of the solvent, <sup>250</sup> ml of freshly deionized distilled water was heated in a microwave oven for 10 minutes at maximum power. <sup>A</sup> 700 watt Panasonic microwave was used for this purpose. Three flasks, Rotavapor, thermometer, reagent grade chloroform, Whatman No.41 filter paper, and MettlerÆ 120 weighing scale was the equipment used. The test packages used in this experiment were exactly similar to the ones used in the previous experiment. FDA 21CFR Ch.1 176.170 was referred to as a reference.

The extraction was carried out with the same equipment, using the same methodology as in previous case. The only difference was the heating of the solvent was done in a microwave.

## RESULTS AND DISCUSSION

4.1 RESULTS:

4.1.1. Conventional Oven Results

According to FDA 21CFR Ch.1 177.1630 "polyethylene pthalate polymers" and <sup>1</sup> 76.1 70, extraction was carried out on the test packages with contact surface being CPET. The solvent used for extraction was distilled deionized water. Two hundred and fifty ml of solvent was subjected to conventional oven heat for 2 hours at 250°F. The results showed that the square package with dimensions 3-3/4" x 3-3/4" x 2-5/8" (96.2mm <sup>x</sup> 96.2mm <sup>x</sup> 67mm) and total surface area in contact with the solvent of 29.0 square inches, had total extractives as shown in table 4-1. The total extractives obtained by evaporating the solvent were 0.7 milligrams, that is, 0.024 mg/square inch of contact surface. Whereas the total chloroform soluble extractives were 0.6 milligrams, that is, 0.020 mg/square inch of contact surface. Temperature and volume of the solvent after 2 hours at  $250^{\circ}$ F in the oven were 154 $^{\circ}$ F and 180 ml.

The rectangular package, with dimensions 5-1 1/16" x 3-3/4" x 1-3/8" (145.5mm <sup>x</sup> 95.5mm <sup>x</sup> 35mm) and total surface area in contact with the solvent of 36.2 square inches had total extractives as shown in table 4-1 . The total extractives obtained by evaporating the solvent were 1.0 milligrams, that

is, 0.027 mg/square inch of contact surface. Also the total chloroform soluble extractives were 0.8 milligrams, that is, 0.022 mg/square in of contact surface. Temperature and volume of the solvent after 2 hours at 250°F in the oven were 150°F and 160 ml respectively.

#### 4.1.2. Microwave Oven Results

The microwave testing for the test package was carried out with square and rectangular packages. Identical methodology was followed as in the previous case except the mode of heating. Instead of heating the solvent at 250°F for 2 hours, the solvent was heated in a microwave for 10 minutes at maximum power in a 700 watt microwave.

The square package with dimensions 3-3/4" x 3-3/4" x 2-5/8" (96.2mm <sup>x</sup> 96.2mm <sup>x</sup> 67mm) and total surface in contact with the solvent of 29.0 square inches, had total extractives as shown in table 4-1 . The total extractives obtained by evaporating the solvent in a microwave oven were 0.9 milligrams, that is 0.031 mg/square inch of contact surface. The total chloroform soluble extractives were 0.5 milligram, that is 0.017 mg/square inch of contact surface. Temperature and volume of the solvent after 10 minutes at high power in the microwave were 220°F and 203 ml respectively. 44

The rectangular package with dimensions 5-11/16" x 3-3/4" x 1-3/8" (145.5mm x 95.5mm x 37mm) and total surface in contact with the solvent of 36.2 square inches, had total extractives as shown in table 4-1 . The total extractives obtained by evaporating the solvent were 1.2 milligrams, that is 0.033 mg/square inch of contact surface. Also the total chloroform soluble extractives were 0.7milligrams, that is 0.019 mg/square inch of contact surface. Temperature and volume of the solvent after 10 minutes at high power in the microwave were 217°F and 201 ml respectively.

In line with the FDA requirements, after two pilot readings, three packages were subjected to the tests in both environments. This meets FDA requirements for sample size in determination of global migration in CPET material according to T. Begeley (1987) and Food and Drug Administration (1976). Complete results are listed in table 4-2.

# Table 4-1. Summary of results of extraction testing. Microwave Oven Heating:



# Table 4-2. Results of extraction testing.



 $\overline{X}$  = EX/N, where X is the symbol for arithmetic mean, EX is the sum of the values and <sup>N</sup> is the total number of samples.

#### 4.2 DISCUSSION

## 4.2.1. Inorganic Migrants

From the results it is clearly evident that the packages meet or better the limit established by the FDA on global extractives for polyethylene terephthalate material. Even though the test package is not meant to be used in the conventional ovens, the chloroform soluble extractives fall under 0.02 mg/ square inch of contact surface, the upper limit established by the FDA. It should be noted that though the package is adequate from the point of view of extraction, the physical properties, such as thickness and low crystallinity of the material prevents it from being totally dual ovenable. On continuous exposure of 2 hours at  $250^{\circ}$ F to the conventional oven heat, curving of the inside edges is observed, making the appearance of the package unesthetic.

It is obvious that the inorganic migrants that are chloroform insoluble, cannot be the monomers that would migrate from within the material. Thus the inorganic migrants can be attributed to surface contaminants, permeating or evaporating into the solvent by means of absorption or desorption. These migrants are usually non volatile and are affected by high temperature generated in the microwave by the solvent. They may originate from adhesives, coatings or other surface treatments that the material could have undergone.

This migration, being a surface phenomenon, is more strongly affected by the high solvent temperature and thus the inorganic or chloroform insoluble extractives are more in case of microwave heating of the solvent rather than the conventional mode of heating.

#### 4.2.2 Organic Migrants

The chloroform soluble extractives obtained through microwave heating of the solvent are even less than the ones obtained by conventionally heating the solvent. This is due to the higher temperature observed during microwaving. The higher solvent temperature induces the CPET material to crystallize more than its inherent 12%. This severely limits the migration of low molecular weight monomers to the solvent. Some of these monomers are polymerized and incorporated into the polymer chain, others may become more active and try to migrate to the solvent phase but as the results indicate, their number is low. Thus it can be assumed that the low level of organic, chloroform soluble, extractives are made up of unpolymerized monomers such as EG, TA, DEG, BHET, MHET and cyclic compounds, which may be the unreacted products of polymerization reaction of the polyester as shown in figure 4.1. If interest exists, the extractives can be analyzed further to obtain the exact quantities of specific chemicals present in the extractives.



$$
-[-0- (CH2)2-COO polyethylene terephthalate
$$

Figure 4. <sup>1</sup> Polymerization reaction and structure of PET

The CPET material is unique in the sense that there is no need for additives such as antioxidants, antifogging agents, catalysts etc., which could migrate at such high temperatures. This is explained by C. Bishop and S. Dye (1982) in their research of migration of plasticizers out of plastics under microwave heating. They have obtained weights of migrants and cited data from studies on harmful effects of plasticizers on animal tissue. It was found that increased temperatures and aging of polymer resin both tend to enhance the migration of plasticizers, monomers and other additives. This test was done on PVC, used as a wrap around material for various products and the plasticizer in question was Di-2-ethylhexyl pthalate (DEPH).

In the case of CPET no plasticizer is used in the manufacturing process and due to lack of crystallinity in the PVC material at higher temperatures, the case cannot be viewed as an exact parallel to the one in this study.

## RECOMMENDATIONS AND CONCLUSION

<sup>A</sup> new methodology was devised to determine the effect of microwave heating of the solvent on total extractives. <sup>A</sup> CPET and paper package was used with CPET being the food contact surface. Water was selected as a solvent and 250 ml of distilled deionized water was heated in a 700 watt microwave for 10 minutes at high power. The test was performed on two package shapes and the results were compared with those obtained by heating the solvent in a conventional oven.

It was found that the amount of chloroform-soluble extractives was less for the packages subjected to microwave heating. The total extractives in weight obtained by microwave heating were comparable to the ones obtained by conventional oven heating.

The CPET material meets or betters the limit set by the FDA for chloroform soluble extractives at 0.02 mg/square inch of contact surface irrespective of the mode of heating the solvent as shown in table 5-1.

# Table 5-1. Summary of results of extraction testing.

# Microwave Oven Heating:



This study investigated only one specific type of polymer (CPET), using only one kind of food simulant. There is a need for further research. Some of the areas that can be considered for further research include, considering other materials for microwave-only packaging and comparing the extractives in a similar way. Materials in which crystallization does not take place may behave differently to high temperature microwave heating of the solvent.

Different materials should be tested for specific additives, such as, antioxidants, colorants, plasticizers, catalysts, etc., along with monomers. Some studies have already been carried out in this area by C. Bishop and A. Dye (1982) and J. Giacin and A. Brzozwska (1985), but what remains to be seen is the effects of microwaving on these particular migration phenomenons.

The FDA, EEC and other regulating agencies must undertake extensive research to determine the toxicity of plastic components at various concentrations, and set limits accordingly. It is understood that selective migration may take place during microwave heating which would necessitate establishing limits for specific migrating species.

Along with packages, plastic cookware should be tested, and directions for use specified clearly. Though in its infancy, microwave cooking has come a long way and is going to set trends for future food processing and designing research. It is necessary that laws are enacted that pertain specifically to the packages and cookware that are to be subjected to microwave cooking or baking.

Microwave cooking of fatty foods pose a different set of problems, as these foods take a longer time to cook. C. Vom Bruck, K. Figge and F. Rudolph (1981) studied the interaction between food components and the packaging material. It is more intricate, involving stress cracking of the material which may lead to higher migration, n-heptane is specified by the FDA as a solvent. Fatty foods are defined as foods which can take up more than 100mg of antioxidant under test conditions of polyethylene film. Microwave cooking of fatty foods and its effects on migration should be studied in future as more meats and oils are used in refrigerated/frozen microwave ready entrees.

Migration also plays a major role in imparting foreign taste and odors to the food that is in contact with the material in question. The effects of microwaving on this phenomenon is yet unknown. Analytical methods are present that can detect and evaluate the presence of this migrants as shown by D. Wyatt (1986). Tests should be performed to study the change in their quantity and composition under microwave heating or cooking of foods.

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Microwave cooking and reheating is carried out in many packages. This procedure should be simulated in the labratory and change in quality of the product evaluated. This can be carried out by calculating migrants after repeated microwave heating of different solvents. Elevated temperatures and accelerated tests should be performed to study the migrants under abusive conditions. Such tests must be performed before approving microwave cookware.

Energy savings, along with savings of time are the main aspects contributing to the success of microwave cooking. Though some studies have been done, more research can be carried out to find the effects of direct energy absorption by food. It is also known that various materials are transparent to microwave radiation upto varying extents. Better use of microwave energy is possible if data is known on permeability of various materials to the microwaves as shown by a study "Ovenable paperboard saves time, energy in tests with microwave and other ovens" in Quick Frozen Foods (1980).

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