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I am submitting herewith a thesis written by David Ray Perrin entitled "Evaluation of Methods for Determining Moisture in Snap Beans Using Microwave and Conventional Oven Drying." I recommend that it be accepted in partial fulfillment of the requirements for the degree of Master of Science, with a major in Agricultural Mechanization.

Luther R. Wilhelm

Luther R. Wilhelm, Major Professor

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EVALUATION OF METHODS FOR DETERMINING MOISTURE IN SNAP BEANS USING MICROWAVE AND CONVENTIONAL OVEN DRYING

A Thesis

Presented for the

Master of Science

Degree

The University of Tennessee, Knoxville

David Ray Perrin

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ABSTRACT

The purpose of this study was to identify both a rapid method for determining moisture and a standard oven drying procedure for determining moisture in snap beans. Parameters of interest were sample size, sample preparation, sample condition (fresh vs. frozen), drying time, oven pressure, and a comparison of microwave, vacuum, and convection oven drying.

The experiment was divided into four phases, each concentrating on selected parameters. The results of one phase determined parameter values used in later phases. Two convection ovens, two vacuum ovens, and one microwave oven were used. For the convection and vacuum oven drying, treatment effects were combinations of drying temperature and oven pressure. Treatment effects for microwave drying were combinations of sample size and power setting (energy level) coupled with drying time. Fresh and frozen samples were evaluated in all three types.

For convection and vacuum oven drying, oven pressure, drying temperature and sample condition had little effect on indicated moisture content for the ranges tested. However, indicated moisture content was more sensitive to temperature than any other factor. Samples used in the microwave oven had to be ground in a food processor before drying. The indicated moisture contents given by the microwave oven were significantly affected by sample size, sample condition, and power setting. The larger samples and higher power settings produced

higher indicated moisture contents. Frozen samples consistently gave higher indicated moisture contents than fresh samples.

For determination of moisture in snap beans, convection ovens at 100°C or vacuum ovens at 100°C with absolute pressures between 760 and 60 mm of mercury gave the same results. Both methods required a 24-hour drying period. Indicated moisture contents given by drying 10-gram samples in the microwave oven for 12 minutes were significantly different than convection or vacuum dried samples at the 95 percent level of probability. Even though they were statistically different, the moisture contents given by the three methods were within 1 percent of each other. Thus, the great time reduction from 24 hours to 12 minutes offered by microwave drying has potential for moisture determination in snap beans.

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FRANKLIN FOREST

CHAPTER I

INTRODUCTION

I. BACKGROUND

The determination of moisture in fruits and vegetables is a necessary, but difficult and time consuming measurement. Water is the principal constituent of all raw foods, and is also an important structurizing constituent of processed foods. Therefore, it is essential for the food industry and researchers to have rapid, relatively accurate and simple methods for moisture determination (Karmas, 1980).

In order to determine moisture content by traditional methods, all the moisture must be removed from a product without burning, or in any way altering the dry matter content. The reference cited most often for methods of moisture analysis is the "Official Methods of Analysis" published periodically by the Association of Official Analytical Chemists (A.O.A.C., 1975). Applicable methods for moisture measurement in fruits and vegetables, however, are not discussed. The A.O.A.C. (1975) does include methods of analysis for dried fruits and for canned and frozen vegetables. Makower et al. (1946) discussed moisture measurement as it applied to dehydrated vegetables.

Even though these methods are not directed at high moisture level fruits and vegetables, some of the procedures involved could be used to develop a method that would give accurate, comparative results. Wilhelm (1979) indicated a relatively simple and reliable method of

moisture determination was needed for comparison of research data with fruits and vegetables.

Presently, there is no single method being used as a standard by researchers. Researchers would benefit from the development of a standard oven drying procedure in the area of physical properties. More importantly, a rapid method of analysis for moisture determination in fruits and vegetables is needed. Since "time is money," a rapid moisture check could be of immense value in both commercial and research applications.

II. OBJECTIVES

This study was conducted to identify a rapid method for determining moisture and a standard oven drying procedure for determining moisture in snap beans.

Specific objectives were:

1. To evaluate the performance of microwave energy for the rapid moisture determination of snap beans. Parameters considered were:
   A. Sample preparation,
   B. Sample size,
   C. Drying time, and
   D. Intensity of microwave energy.
2. To identify a potential standard oven drying method for moisture determination in snap beans. Parameters considered were:

A. Drying temperature,

B. Oven pressure, and

C. Sample condition (fresh vs. frozen).

CHAPTER II

REVIEW OF LITERATURE

I. IMPORTANCE

The snap bean (Phaseolus vulgaris) is but one of numerous high moisture level vegetables. Gould (1977) indicated that snap beans averaged 88.9 percent water. The snap bean is an important vegetable for human consumption. The high annual consumption of snap beans causes them to be economically important to the commercial processor.

The Tennessee Agricultural Statistics in 1978 indicate that 80 percent of the snap beans harvested in Tennessee were for processing. This brought an income of over $3,000,000 to farmers in that market alone (Tennessee Farm Facts, 1979).

Much research has been conducted on snap beans. Moisture content is usually a factor of interest to the researcher. Oven drying procedures are normally used to determine the moisture level in snap beans. Makower et al. (1946) concluded that oven drying under existing industrial standards was "not satisfactory from the standpoint of either precision or accuracy."

According to Van Arsdel et al. (1973), the magnitude of the "moisture content" of foodstuff or its raw materials depends greatly on the method used for moisture determination (i.e., all definitions of this term are operational). Many different methods of determination have been proposed for one purpose or another.

II. METHODS USED FOR MOISTURE DETERMINATION

Of the numerous methods available to determine moisture in vegetables, Makower et al. (1946) suggested that the "vacuum oven method" is the most important because it is used currently as a reference for the calibration of other methods. Other methods used for determining moisture content in fruits and vegetables are: (1) electrical, (2) toluene distillation, (3) Fisher volumetric, (4) nuclear magnetic resonance, (5) dichromate oxidation, and (6) infrared radiation.

Choice of a method is based on the importance attached to accuracy, precision or reproducibility, time required for a determination, availability of necessary equipment, degree of skill or training required and several other factors according to Stitt (1958).

From research conducted by the U.S. Department of Agriculture, the recommended method for determining the moisture content in dried fruits is not sufficiently rigorous to ensure agreement among different testing laboratories (Makower et al., 1946). The method questioned requires the material to be ground in a food grinder and heated in a vacuum oven at 70°C for 6 hours at a pressure not exceeding 100 mm of mercury.

Makower et al. (1946) did outline conditions for drying potatoes, carrots, cabbage, and onions. For either vegetable, the sample had to be prepared by grinding over 100 grams of material in a Wiley mill and passing it through a U.S. 10-mesh sieve. Then 25 gram portions were reground and passed through a 40-mesh sieve. Drying was accomplished

in a vacuum oven maintained at a pressure of 5 mm of mercury or less. The drying times for a permissible error in the determination of 0.1 percent were as follows: cabbage at 60°C for 22 to 40 hours; carrots at 70°C for 29 to 35 hours; onions at 60°C for 15 to 45 hours; and potatoes at 70°C for 43 to 67 hours. To dry large numbers of samples or samples of different vegetables, he suggested drying at 70°C for 40 hours. Luh and Jasper (1975), citing the 1970 edition of the A.O.A.C., suggested the drying of a sample of specified size in a vacuum oven for 6 hours, or until there was no further weight loss.

Many different and similar procedures are listed by various publications. A summary of several possible time, temperature, and vacuum combinations recommended for various products is shown in Table 1.

Wilhelm (1979) conducted an experiment using convection and vacuum ovens to evaluate the effects of temperature, sample preparation, and slight vacuum upon the indicated moisture content of snap beans. Temperatures of 65, 100, 135, and 170°C and absolute pressures of 760, 735, 710, and 660 mm of mercury were used. Three different sample preparation treatments were used: whole pods; pods split lengthwise into two pieces; and pods cut into 25-mm lengths. Drying times were 2, 6, and 24 hours.

He found that the 24 hour drying period was the only time that yielded consistent results. The higher oven temperatures produced significantly higher indicated moisture contents. Oven vacuum and sample preparation had no significant effect on indicated moisture

Table 1. Time, Temperature, and Vacuum Combinations Used for Moisture Measurement of Selected Products

Product	Temperature (°C)	Vacuum or Absolute Pressure	Drying Time	Reference
Cheese	100	<100 mm Hg	4 hr	AOAC, 1975; (16.217)
Egg Solids	98-100	<25 mm Hg	5 hr	AOAC, 1975; (17.007)
Coca bean, butter	100	none	until dry	AOAC, 1975; (13.002)
Flour	130 ± 3	none	1 hr	AOAC, 1975; (14.002)
Potatoes, green beans	70	<100 mm Hg	6 hr	Van Arsdel et al., 1973
Cabbage	60	<100 mm Hg	16 hr	Van Arsdel et al., 1973
Onions, leafy vegetables	60	<5 mm Hg	30 hr	Van Arsdel et al., 1973
Dried fruits	70 ± 1	<100 mm Hg	6 hr	AOAC, 1975; (22.013)
Animal feed, plants	90-100	<100 mm Hg	5 hr	AOAC, 1975; (7.003)
Frozen vegetables	105	forced draft	until dry	AOAC, 1975; (32.048)
Canned vegetables	70 ± 1	<50 mm Hg	2 hr	AOAC, 1975; (32.004)
Meats	100-102	none	16-18 hr	AOAC, 1975; (24.003)
No product specified	70	26 in. (Hg)	6 hr	Gould, 1977

content when using the 24 hour drying period. However, further study using much higher vacuum levels was recommended.

Recently, microwave oven techniques have been explored for rapid moisture determination of various products (Pieper et al., 1977; Gorakhpurwalla et al., 1975; Becwar et al., 1977; Davenport et al., 1975; Pettinati, 1975; Lee and Latham, 1976; and Hankin and Sawhney, 1978). In 1975 the Association of Official Analytical Chemists adopted a microwave technique (16.001) for a rapid determination of moisture in cheese (JAOAC, 1977).

III. BASIC PRINCIPLES OF MICROWAVES

Microwaves are electromagnetic waves similar to radio, television, and light waves. They transmit energy through space, just as electricity is transmitted through a wire. All electromagnetic waves are characterized by wave length and frequency. Two frequencies have been appointed by the FCC for microwave power generation in commercial and industrial applications. Those frequencies are 2450 megahertz and 915 megahertz. They have respective wave lengths of 12.2 cm and 33 cm (Schiffman, 1976).

Microwaves have many of the characteristics of light waves: they travel in a straight line; they can be generated; and they can be reflected, transmitted, and absorbed. The basic difference between light waves and microwaves is in the materials that reflect, transmit, and absorb them (Pieper et al., 1977).

Microwave power is generated in a special vacuum tube. Presently, there are two types of tubes being used to generate microwaves. They are the klystron and the magnetron tubes. The klystron tube is an expensive higher powered type of microwave generating tube which requires water cooling. The magnetron tube is a lower powered microwave generating tube which is cooled by air. It has proven to be a very economical method of generating microwave energy (Davenport et al., 1979).

When microwave energy is generated in an oven, the electromagnetic field within effectively changes direction millions of times per second. The molecules comprising the sample being treated attempt to shift their position to align themselves with this ever changing polarity of the electromagnetic field. The intermolecular friction between these millions of molecules oscillating about their axes produces heat throughout the sample being treated. Thus, a material heated by microwave energy is more or less uniformly heated throughout, instead of being heated from the inside out (Pieper et al., 1977).

Since a material is heated throughout it is possible to speed up laboratory moisture analysis. It also reduces empirical error introduced by sample preparation required for conventional heat methods (Pieper et al., 1977). The time required to heat a sample depends on the amount of heat required and the weight of the sample. The factors influencing heat requirements are: initial product temperature; size; consistency; specific heat; shape; and dielectric properties of the test product (Schiffman, 1976).

Instructions for heating prepared items in a microwave oven may present problems. Gerling (1978) stated that microwave ovens vary from model to model. The variations occur when one compares indicated power to effective power output with loads of different size. He emphasized that a simple calculation of power output can be misleading. One test he mentioned to measure power output involved the heating of 160 grams of water in polystyrene cups. The power output of the microwave oven for each load could be calculated then knowing the temperature rise of the load, the weight of the load, and the time of the test. The results of this method were comparable to other methods used for measuring power output.

IV. APPLICATIONS OF MICROWAVE ENERGY TO MOISTURE MEASUREMENTS

Moisture level is a factor commonly used to determine the proper harvest date of sweet corn for processing. The two standard methods of determining moisture level in corn are drying 72 hours in a hot air oven (ASAE) or by drying 24 hours in a vacuum oven (AOAC).

Becwar et al. (1977) compared microwave heating with the vacuum oven method and got comparable results. The results were within 1 percent of results obtained by the vacuum oven method. The optimum heating time for 10-gram samples placed on petri dishes was 3 minutes. Samples were prepared by blending whole kernels of corn for 2 minutes in a blender.

In the study by Becwar et al. (1977), whole kernels were not used because they splattered in the oven. Davenport et al. (1979) also found that microwave drying of whole kernels of corn was impractical, but for another reason. They obtained significant variations of indicated moisture (4.02 percent variation for microwave drying vs. 1.01 percent variation for conventional drying).

Davenport et al. (1979) found that deformed corn (corn mashed by a hammer) dried by microwaves compared well with conventional oven drying. They dried 20-gram samples for 25 minutes. Variations for the microwave and oven dried samples were within 1 percent of one another.

Fanslow and Saul (1979) combined microwave energy and unheated air to dry field corn. They found that the microwave power used to dry grain caused a considerable reduction in processing time. Since the kernels were heated throughout, case hardening of the kernels was eliminated. They concluded that there was a practical limit to the speed at which corn could be dried. Beyond that limit there was enough swelling of the kernels to lower the market grade.

Gorakhpurwalla et al. (1975) studied high moisture grain drying using a microwave method in conjunction with heated forced air and a rotating sample holder designed to reduce reflections and prevent thermal runaway conditions. They obtained an accuracy of 1 percent in drying corn and sorghum grain with initial moistures of about 35 percent.

Pieper et al. (1977) used a specially designed microwave unit, with an internal balance, a recirculating water system, and a variable power control for adjusting microwave intensity, to develop a microwave technique for a rapid determination of moisture in cheese. Determinations could be made in only 2.25 minutes of drying. Results compared favorably with existing AOAC methods. Ten gram samples were found to be optimum size. Samples were prepared according to AOAC method 16.216 and placed on pyrex petri dishes before heating.

Pettinati (1975) developed a new, rapid procedure for moisture determination in meat. Five gram samples were heated 2.5 minutes in a 1000 watt domestic-type microwave oven. After heating, the residues were exposed 1 minute in the stream of the oven chamber air blower, then covered and weighed. Results of the test were comparable with AOAC method 24.003(b). The meat samples were dispersed with a mixture of sodium chloride, ferrous oxide, and sand. The addition of sand eliminated splattering during treatment. The addition of sodium chloride and ferrous oxide, a known strong absorber of microwave energy, resulted in accelerated drying times (from 15 minutes to 2.5 minutes).

Lee and Latham (1976) developed a rapid method for moisture determination of canned pet food using an Amana commercial microwave "Radarange" oven, model RC14. The complete drying procedure only required 3.5 minutes. Less than 2 minutes of actual microwave heating were required. A 10-gram homogenized sample was placed between two pieces of filter paper (Whatman No. 40). The sandwiched sample was heated for 30 seconds; air dried for 15 seconds; lifted and turned

over, and heated for 40 seconds; then turned back over and heated 20 additional seconds.

The moisture level in soil is usually determined by heating samples 12 to 24 hours at 110°C (Davenport et al., 1979). Hankin and Sawhney (1978) and Davenport et al. (1979) performed tests for soil moisture determination using microwave energy. The results of their experiments were contradictory. Hankin and Sawhney (1978) found no significant difference between heating soil samples 6 minutes by microwaves versus hot air heating at 110°C for 18 hours. Davenport et al. (1979) found that microwave drying consistently removed less moisture than the conventional oven method used.

Davenport et al. (1979) dried samples of 20 and 30 grams placed on paper plates for 12 to 72 minutes depending on the soil type and oven setting. The oven settings used were 100, 70, and 30 percent of total power output.

Hankin and Sawhney (1978) found that 10-gram soil samples were adequately dried in 6 minutes. In this experiment, two pieces of filter paper (Whatman No. 1) were used to make containers for the soil samples. The filled containers were placed on paper towels, loaded into the oven, and treated for 3 minutes. The containers were turned over then and treated for an additional 3 minutes. The containers were removed from the oven, and exposed to air flow either by a fan or fume hood for 30 seconds.

Microwave energy has been used successfully as a time-saving method for preparing specimens of pine cross-sectional disks when the

disks were of moderate thickness (Illingworth and Klein, 1977). The specimens were dried from a moisture content of 40 percent. Disks 1-inch thick were dried in 45 minutes at 100 percent power without checking.

McAlister and Resch (1971) dried 1-inch ponderosa pine lumber with a combination of microwave power and hot air. They concluded that 1-inch boards, having a 50 percent moisture content, could be dried without defects in less than 3 hours.

Barnes et al. (1976) indicated that a prototype continuous drying system for lumber (clear grade of Douglas-fir and hemlock) utilizing microwave energy had been developed. The system dried lumber 30 times faster than conventional kiln drying.

In an experiment conducted by Darrah et al. (1977), forage samples treated with microwave energy had greater heat damage than samples dried at 50°C. They noted that microwave treatment may be an acceptable method of stopping respiration in fresh tissue without causing significant amounts of heat damage, but microwave pretreatment of forage samples intended for chemical analysis should be used with caution.

Reveron et al. (1971) found that microwave drying of lamb carcass samples was an alternative to freeze drying in the comparative slaughter technique for the evaluation of animal foods. They heated 50 gram amounts of carcass placed in shallow pyrex dishes 60 to 80 minutes in a microwave cooker.

CHAPTER III

PROCEDURE

I. GENERAL INFORMATION

Due to the limited number of ovens and an intermittent supply of fresh snap beans, the experiment was divided into four phases. The time required to perform a sufficient number of tests involving all combinations of factors with the resources available was prohibitive. Therefore, each phase concentrated on certain parameters. The results of one phase were used to determine parameter values used in later phases.

Five separate ovens were used--two vacuum ovens, two convection ovens, and one microwave oven. However, every oven was not used in every phase.

Beans were obtained from The University of Tennessee Plant and Soil Science Farm, The University of Tennessee Plateau Experiment Station, and from independent growers in the Cumberland Plateau region. Some of the beans were hand-picked, but most were machine harvested. The beans were sorted according to size. Only pods of a number 5 sieve size were selected for testing.

Analyses of variance, orthogonal contrasts, Duncan's Multiple Range Tests, and simple statistics were obtained through use of the SAS 79 package available at The University of Tennessee Computing Center.

II. EQUIPMENT AND MATERIALS

The following equipment and materials were used:

1. Oven (CQ), Precision Scientific Company convection oven. Catalog No. 1244, 230 volts, 18.0 amps, and 4100 watts. Forced air convection with 1.36 m^2 shelf space.
2. Oven (CL), Precision Scientific Company convection oven. Catalog No. L470, 115 volts, 6.0 amps, and 700 watts. Natural air convection with 0.28 m^2 shelf space.
3. A. Oven (FV), Fisher Scientific Company Isotemp vacuum oven. Model No. 281, 120 volts, and 8.5 amps.
   B. Welch Duo-Seal vacuum pump, Sargent Welch Scientific Company. Model No. 1400. Guaranteed pressure 13.3 MPa.
4. A. Oven (NV), National Appliance Company vacuum oven. Model No. 5851.
   B. Welch Duo-Seal vacuum pump, Sargent Welch Scientific Company. Model No. 1402. Guaranteed pressure 13.3 MPa.
5. Amana Touchmatic II Radarange Microwave Oven, Amana Refrigeration Inc., Amana, Iowa. Model No. RR-10A, 2450 MHz, 120 volts, and 750 watts maximum power output (modified).
6. Food Processor, Farberware Inc., Bronx, New York. Model No. 286.
7. Corning Pyrex Lab Glassware, Springs, Oklahoma. 100 x 10 mm dishes.
8. Aluminum cans with lids. 5.5 x 9 cm.

9. Balance, Metler E200, Metler Instrument Corp., Hightstown, New Jersey. No. 708556, maximum 200 g.
10. Desiccant, Drierite Anhydrous Hammand $CaSO_4$, W.A. Hammand Drierite Company, Xenia, Ohio. Size 10-20 mesh.
11. (2) Gas Drying Jars with absorbent, Fisher Scientific Company. Catalog No. 9-204, used in vacuum line between vacuum oven and pump.

III. PHASE ONE (TEMPERATURE AND VACUUM EFFECTS)

In phase one the parameters of interest were: drying temperature and oven pressure. Four separate ovens were used--two convection ovens and two vacuum ovens. The convection ovens were used to dry samples at 100°C, while the vacuum ovens were operated at 65°C and 100°C. Vacuum ovens were operated at absolute pressures of 660 and 60 mm of mercury (88 and 8 kPa). An incomplete block experimental design was used. Treatment combinations considered in the various tests are given in Table 2.

The beans used in each test were frozen. Therefore, the beans had to be placed into cans and covered immediately after removal from the freezer. In every test, five beans each were placed into eight preweighed cans. Pods were broken in half to fit inside the can. Each of the four drying ovens was loaded with two cans after weighing. The cans were removed and weighed after a 24-hour drying period. The weight lost by each sample at the time of weighing was taken as the moisture removed from the beans. Using this weight, the indicated moisture content was then computed.

Table 2. Treatment Combinations (Absolute Pressure and Temperature) for Phase One

Test	Vacuum Ovens				Convection Ovens	
	Oven FV[a]		Oven NV[a]		Oven CQ[a]	Oven CL[a]
	Temperature (°C)	Absolute Pressure (mm Hg)	Temperature (°C)	Absolute Pressure (mm Hg)	Temperature (°C)	Temperature (°C)
1	65	660	100	60	100	100
2	100	660	65	60	100	100
3	100	60	100	660	100	100
4	65	60	100	60	100	100
5	100	660	65	660	100	100
6	65	60	65	660	100	100
7	65	660	65	60	100	100
8	100	60	100	660	100	100
9	65	660	65	60	100	100
10	100	60	65	660	100	100
11	100	60	65	660	100	100

[a]See Equipment and Materials section for an explanation of oven types.

IV. PHASE TWO (FRESH VS. FROZEN)

In phase two, only one parameter was considered: sample condition (frozen vs. fresh). Only the two vacuum ovens were used in these tests. Both vacuum ovens were used to dry samples at 100°C, and they were operated at an absolute pressure of 60 mm of mercuey (8 kPa).

Beans were harvested on two different occasions. From each of the two harvests, one test using fresh beans was run, and three tests using frozen beans were conducted. In every test, five beans were placed into each of eight preweighed cans. Pods were broken in half to fit inside the can. The indicated moisture content was determined by the same procedure as noted in phase one.

V. PHASE THREE (MICROWAVE)

In phase three, only the microwave oven was used. Parameters of interest were: sample size, oven power setting, drying time, and sample condition.

Before any drying tests were run, the microwave oven was checked for power output using different size loads and different power settings. Actual loads used were 100 and 200 grams of distilled water placed in polystyrene cups. The oven was operated at full power and at setting No. 5. The water in the test load, initially at room temperature (22°C) was heated to 77°C. The time required for the temperature to increase from 49°C to 77°C was recorded. Five tests for each sample size and power setting combination were run. One sample per test was

used. The power output of the oven for each load could be calculated then knowing the temperature rise of the load, the mass of the load, and the time of the test.

For the drying tests, the microwave oven was operated at power settings of 4, 6, 8, and 10 (full power). Sample sizes used were 5, 10, 15, and 20 grams. Two different sample conditions were used: frozen and fresh. Drying times varied. They were dependent on combinations of sample size and power setting. An outline of the experiment listing the treatment combinations and drying times is shown in Table 3.

Beans were harvested on three occasions. From each of the three harvests, one replication using fresh beans and one replication using frozen beans were run.

For both fresh and frozen beans, the samples were prepared as follows: approximately 400 grams of pods were introduced into a food processor and ground until a viscous liquid was formed. Original plans were to use a food blender, but in preliminary work the blender did not liquify the beans adequately. As an alternative, a food processor was tested. The food processor liquified the beans satisfactorily. During preliminary work with the food processor, 400 grams of fresh beans were found to be the minimum amount that would liquify satisfactorily. The frozen beans reacted differently in the processor. As a result, 200 grams were sufficient to liquify the frozen beans.

In each drying test the prepared sample was placed on two preweighed pyrex petri dishes. The filled dishes were weighed and placed into the microwave oven.

Table 3. Microwave Experimental Design Including Drying Times for Phase Three[a]

Test	Treatment	Replication 1 Power Setting	Sample Size	Drying Time (minutes) T1	T2	T3	T4
1	4	10	5	4	5	6	7
2	11	8	15	7	10	13	16
3	3	8	5	6	7	8	9
4	6	6	10	8	10	12	-[b]
5	7	8	10	6	7	8	9
6	5	4	10	12	14	16	18
7	10	6	15	10	11	12	13
8	16	10	20	8	9	10	11
9	1	4	5	7	8	9	10
10	14	6	20	12	14	15	-[b]
11	9	4	15	13	15	17	19
12	2	6	5	6	7	8	9
13	12	10	15	6	8	10	12
14	13	4	20	15	18	21	24
15	8	10	10	6	7	8	9
16	15	8	20	7	9	11	13

[a]One of six replications that was run. Other replications had different treatment and test combinations.

[b]In these two treatments, no further weight loss occurred after three time intervals.

The dishes for each treatment were removed and weighed at various time intervals as indicated in Table 3. The time intervals used were based on preliminary work with the microwave oven. The samples were checked at short intervals after the initial drying period to determine when there were no further weight losses. Various combinations were tested, and the time intervals used provided the most accurate and reliable results.

The weight loss by each sample at the last time of weighing was taken as the moisture removed from the beans. Using this weight, the indicated moisture content was computed.

VI. PHASE FOUR (VACUUM, CONVECTION, AND MICROWAVE)

In phase four both vacuum ovens, one convection oven, and the microwave oven were used. There were two areas of interest in phase four. They were: (1) the effect of oven pressure on the indicated moisture content and (2) a comparison of the indicated moisture content given by the four ovens. An incomplete block experimental design was used. Treatment combinations considered in the various tests are given in Table 4. For the microwave oven a complete block experimental design was used. An outline of the design is given in Table 5.

Both vacuum ovens and the convection oven were used to dry samples at 100°C. The vacuum ovens were operated at absolute pressures of 760, 660, 360, and 60 mm of mercury (101, 88, 48, and 8 kPa). A 24-hour drying period was used in the convection and vacuum ovens.

Table 4. Treatment Combinations (Oven Pressure) for Phase Four Convection and Vacuum Oven Part

Test	Treatment[a] Absolute Pressure in Vacuum Oven (mm Hg) 660	360	60	760	Convection Oven Atm. Pressure
1		FV[b]		NV[b]	CQ[b]
2		NV	FV		CQ
3	NV			FV	CQ
4	NV		FV		CQ
5	NV	FV			CQ
6			NV	FV	CQ
7		NV		FV	CQ
8		FV	NV		CQ
9	FV			NV	CQ
10	FV		NV		CQ
11	FV	NV			CQ
12			FV	NV	CQ

[a]All ovens were operated at 100°C.

[b]Designates the particular oven used as described in Equipment and Materials section.

Table 5. Microwave Experimental Design in Phase Four (Tests with Frozen Samples for Comparison with Vacuum and Convection Oven Drying)

Test	Sample	Treatment[a]	Replication
1	1	6	1
			2
			3
			4
	2	6	1
			2
			3
			4
	3	6	1
			2
			3
			4
	4	6	1
			2
			3
			4
2	1	6	1
			2
			3
			4
	2	6	1
			2
			3
			4
	2	6	1
			2
			3
			4
	4	6	1
			2
			3
			4

[a]Treatment 6 is the same as in Table 3.

In each drying test, four cans were loaded into the ovens. The cans were weighed before filling. They were filled with 5 frozen pods broken in half. The filled cans were reweighed. Four different sources of beans were used for sample selection. Each can contained a sample from only one source, therefore, all sources were represented in every oven in each drying test.

After drying, the cans were removed and weighed. The weight loss by each sample at the time of weighing was taken as the moisture removed from the beans. Using this weight, the indicated moisture content was computed.

The microwave oven was operated at power setting 6. Ten-gram samples were used. The samples were prepared as follows: approximately 200 grams of frozen pods were introduced into a food processor and ground until a viscous liquid was formed. The samples were placed on preweighed pyrex petri dishes. Then, the filled dishes were reweighed. The microwave oven was loaded with two dishes in every test. The dishes were removed and weighed at 8-, 10-, and 12-minute intervals after loading. The samples were checked at the 8- and 10-minute intervals to ensure there was no further weight loss at the 12-minute interval. The weight loss by each sample at the time of weighing was taken as the amount of moisture removed from the beans. Using the weight at the 12-minute interval, the indicated moisture content was computed.

CHAPTER IV

RESULTS AND DISCUSSION

I. PHASE ONE (TEMPERATURE AND VACUUM EFFECTS)

The indicated moisture content (wet basis) was evaluated by oven drying temperature, oven pressure, the particular oven used, test differences, and vacuum and convection oven drying differences. The analyses and orthogonal contrasts are given in Table 6.

The overall treatment effects (which included drying temperature, oven pressure and convection versus vacuum oven drying) were significant at the 95 percent level of probability. However, partitioning the treatment effects resulted in no significant differences for either temperature and oven pressure ($P = 0.95$).

Wilhelm (1979) found significant differences between drying temperature using temperatures of 65, 100, 135, and 170°C. He concluded that the 135 and 170°C temperatures accounted for the differences.

There was no significant difference between the two vacuum ovens or between the two convection ovens ($P = 0.95$). There was a significant difference between convection and vacuum oven drying ($P = 0.95$). There was no factor which explained that difference.

Samples used in each test were from different sources, therefore, test-to-test variations were significant. The treatment combinations were arranged to permit removal of the effect of test-to-test variations in the statistical analysis.

Table 6. Analyses and Orthogonal Contrasts for Phase One

Source	DF	Type I SS	F Value	PR > F	DF	Type III SS	F Value	PR > F
Test	10	0.02641323	64.93	0.0001	10	0.02408081	59.20	0.0001
Treatment	4	0.00065941	4.05	0.0051	3	0.00013162	1.08	0.3644
Oven	2	0.00010632	1.31	0.2771	2	0.00010632	1.31	0.2771

Contrast	DF	SS	F Value	PR > F
Temp 65 vs 100	1	0.00010147	2.49	0.1187
Pressure 660 vs 60	1	0.00005405	1.33	0.2529
Oven FV vs NV	1	0.00003575	0.88	0.3517
Oven CQ vs CL	1	0.00007056	1.73	0.1920
Inter vacuum	1	0.00000323	0.08	0.7789
Convection vs vacuum[a]	1	0.00047376	11.55	0.0011

Source	DF	Sum of Squares	Mean Square	F Value	PR > F	R-Square	C.V.
Model	16	0.02717896	0.00169868	41.76	0.0001	0.903944	0.7025
Error	71	0.00288812	0.00004068	Std Dev		M24 Mean	
Corrected total	87	0.03006707		0.00637781		0.90783963	

[a]This contrast was taken from a less sensitive model with the variable oven excluded.

Since drying temperature was non-significant, 100°C was the only temperature used in phases two and four. Even though oven pressure was non-significant, it was considered later since there were many pressure combinations untested.

II. PHASE TWO (FRESH VS. FROZEN)

The indicated moisture content (wet basis) was evaluated by sample condition (fresh vs. frozen) and the particular oven used. Table 7 lists the results for fresh and frozen sample tests. The analyses and orthogonal contrasts are given in Table 8.

Sample condition was not significant at the 95 percent level of probability. It was concluded that either fresh or frozen beans could be used for drying tests provided the beans are frozen as soon as possible after harvest to prevent dehydration.

The vacuum ovens used were not significantly different at the 95 percent level of probability. The vacuum ovens were also found to be non-significant in phase one. Therefore, similar vacuum ovens operating under the same conditions should give similar results with both fresh or frozen beans.

III. PHASE THREE (MICROWAVE)

Microwave Oven Power Output

Figure 1 summarizes the microwave oven power output using 100- and 200-gram loads of distilled water. These tests were run to provide a reference for comparison with other similar microwave units.

Table 7. Means of Comparative Determinations for Percent Moisture in Fresh and Frozen Bean Samples

Sample[a]	Test	Sample Condition	Indicated Moisture[b] (percent)
1	1	fresh	89.78
	2	frozen	89.82
	3	frozen	89.89
	4	frozen	89.47
2	1	fresh	81.78
	2	frozen	81.32
	3	frozen	81.03
	4	frozen	82.52

[a]Sample 1 was picked under normal climate conditions, and sample 2 was picked in an abnormally dry period.

[b]Indicated moisture is mean of 8 determinations per test.

Table 8. Analyses and Orthogonal Contrasts for Fresh and Frozen Beans on Indicated Moisture

Source	DF	Type I SS	F Value	PR > F	DF	Type III SS	F Value	PR > F
Test	7	0.10545465	89.73	0.0001	7	0.10545465	89.63	0.0001
Oven	1	0.00018842	1.12	0.2943	1	0.00018842	1.12	0.2943

Contrast	DF	SS	F Value	PR > F
Sample 1 Fr[a] vs Fz[b]	1	0.00000207	0.01	0.9121
Sample 2 Fr vs Fz	1	0.00001483	0.09	0.7676
Both 1 and 2 Fr vs Fz	1	0.00001398	0.08	0.7741

Source	DF	Sum of Squares	Mean Square	F Value	PR > F	R-Square	C.V.
Model	8	0.10564307	0.01320538	78.57	0.0001	0.9919539	1.5127
Error	55	0.00924398	0.00016807	Std Dev		M24 Mean	
Corrected total	63	0.11488705		0.01296427		0.85700975	

[a]Fresh

[b]Frozen

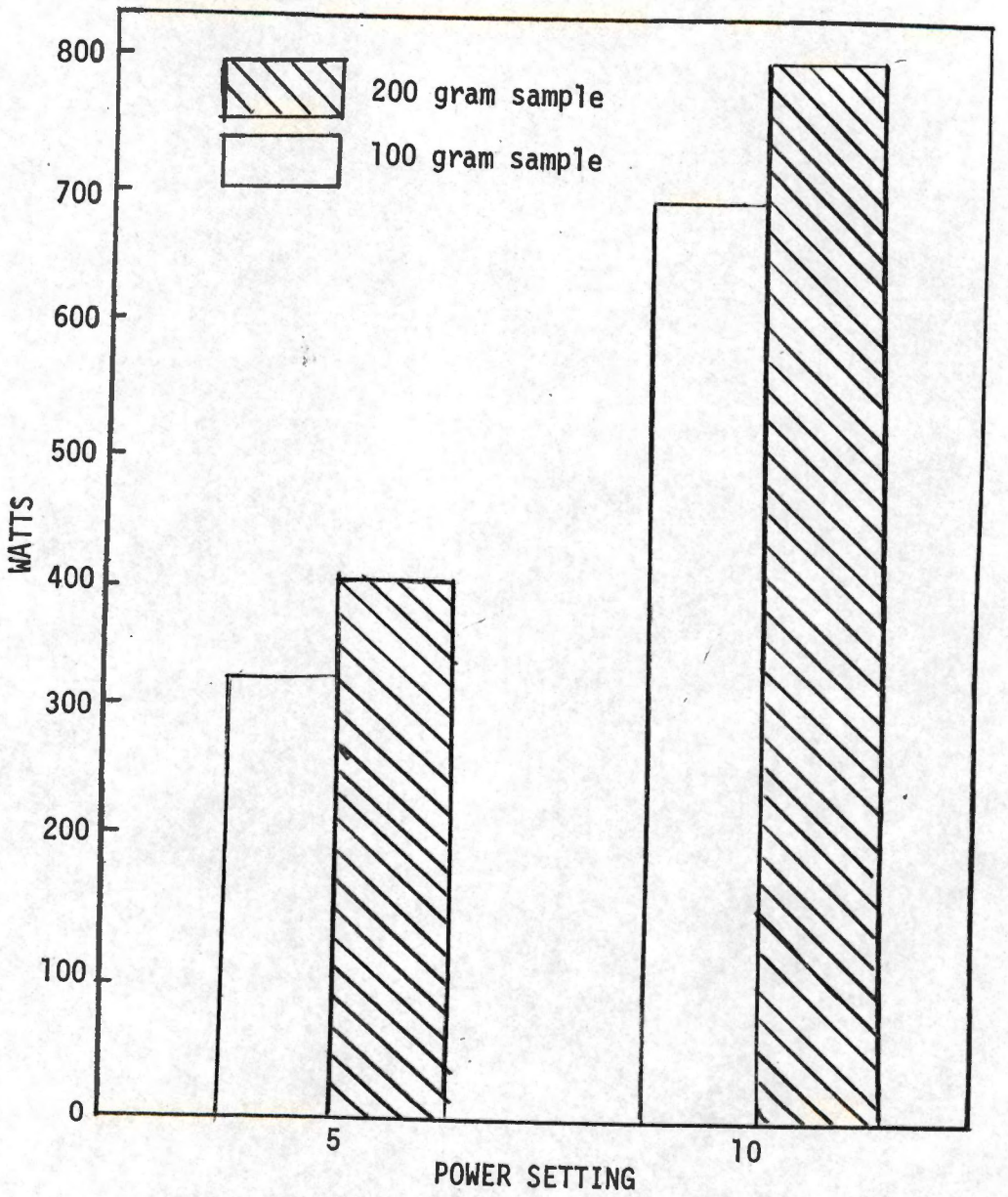


Figure 1. Measured microwave power output to raise the temperature of 100 and 200 gram distilled water samples from 49°C to 77°C; average of five tests.

Microwave Drying Tests

The indicated moisture content (wet basis) was evaluated by treatment effects, sample condition and replicate differences. Table 9 lists the results of each treatment for both sample conditions. The analyses and contrasts are given in Table 10.

Instead of evaluating specific oven power settings and sample sizes, treatment combinations of these two factors were evaluated. Treatment combinations were mentioned earlier in Table 3, page 21.

The overall treatment effects were significant at the 95 percent level of probability. Some treatments were non-significant, but none of the treatments was non-significant when compared with all other treatments. A Duncan's Multiple Range Test and a probability difference option used in conjunction with procedure GLM in SAS 79 were used to identify the least significant treatment. Both tests showed that treatment 6 was similar to more treatments than any other individual treatment, but treatment 6 was significantly different from seven other treatments. Figure 2 shows the sample condition of treatment 6 after drying.

One problem encountered that might account for the treatment differences was burning of the sample during drying. The higher power settings greatly increased the sample area percentage that burned. Figure 3 shows a burned sample (treatment 12) that had been dried at power setting 10 (full power). In every replicate, treatment 12 caused the sample to burn. Except for treatment 1, treatment 12 was significantly different from more treatments (different from 12 others) than

Table 9. Microwave Oven Results of Treatment Effects on Fresh and Frozen Samples

Treatment	Indicated Moisture Content (percent)[a]	
	Fresh	Frozen
1	86.67	87.45
2	86.91	88.01
3	87.83	88.32
4	87.43	88.37
5	87.26	87.79
6	87.50	87.94
7	87.24	87.87
8	87.51	88.16
9	87.27	87.60
10	87.27	87.80
11	87.92	88.06
12	87.93	88.45
13	87.32	87.77
14	87.48	87.86
15	87.58	88.05
16	87.81	88.27

[a]Average of three replications.

Table 10. Microwave Drying Analyses and Orthogonal Contrasts for Fresh and Frozen Snap Beans

Source	DF	Type I SS	F Value	PR > F	DF	Type III SS	F Value	PR > F
Replication	5	0.15207901	3117.82	0.0001	5	0.15207901	3117.82	0.0001
Treatment	15	0.00154234	10.54	0.0001	15	0.00154234	10.54	0.0001
Treatment x Replication	75	0.00174497	2.38	0.0001	75	0.00174497	2.38	0.0001

Contrast	DF	SS	F Value	PR > F
Rep 1(Fr) vs Rep 2(Fr)	1	0.00000169	0.17	0.6781
Rep 1(Fr) vs Rep 3(Fz)	1	0.00193384	198.23	0.0001
Rep 2(Fr) vs Rep 4(Fz)	1	0.00014811	15.18	0.0002
Rep 3(Fz) vs Rep 4(Fz)	1	0.00093055	95.39	0.0001
Rep 5(Fr) vs Rep 6(Fz)	1	0.00010525	10.79	0.0014
Fresh vs Frozen	1	0.00147987	150.67	0.0001

Source	DF	Sum of Squares	Mean Square	F Value	PR > F	R-Square	C.V.
Model	95	0.15536633	0.00163544	167.64	0.0001	0.994008	0.3561
Error	96	0.00093652	0.00000976	Std Dev		M Mean	
Corrected total	191	0.15630285		0.00312337		0.87708747	

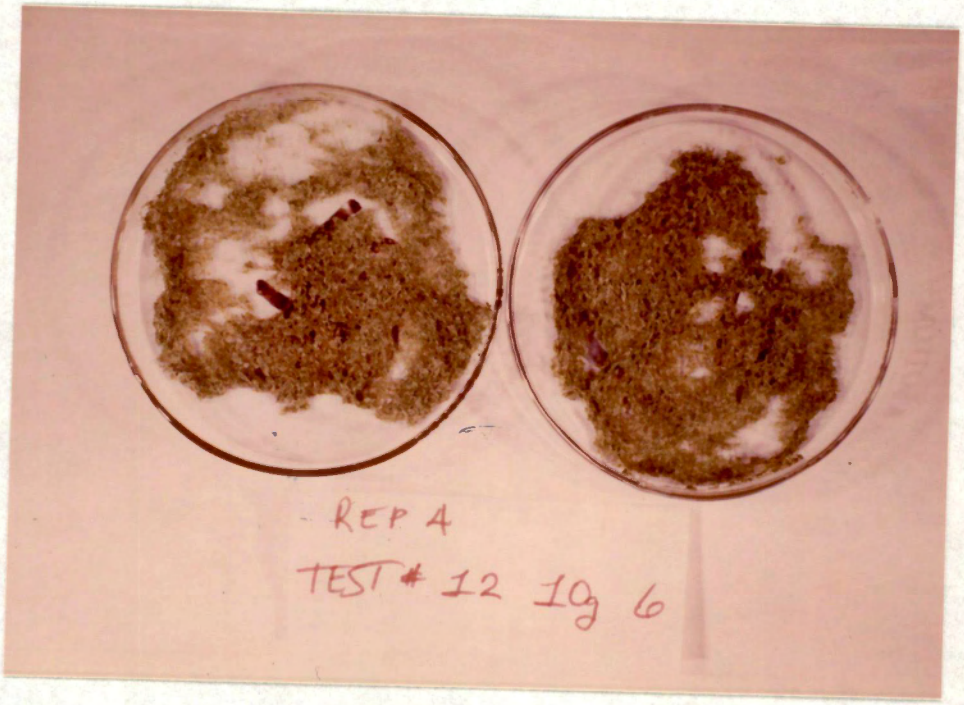


Figure 2. Sample condition of treatment 6 after drying 10 grams at oven setting 6 for 12 minutes.

Note: Black mark is letter on petri dish.



Figure 3. Sample condition of treatment 12 after drying 15 grams at oven setting 10 for 12 minutes.

any other treatment. All treatments using power setting 10 (full power) showed major burning. Treatments using power settings 4 or 6 were not burned, while those with power setting 8 exhibited burning of at least 50 percent of the sample. Figure 4 shows a sample (treatment 13) with no burning dried at power setting 4.

Treatment 1 consistently gave lower indicated moisture levels than the other treatments. Apparently, the drying times were too short to allow complete moisture removal from the sample.

Replicate effects were significant at the 95 percent level of probability. Replicates 1 and 2 were not significantly different at the 95 percent level of probability. The other replicates were significantly different ($P = 0.95$). Samples in replicates 1 and 2 came from the same source. The sample used in replicate 5 came from another source. The samples used in replicates 3, 4, and 6 were frozen. They came from the same sources that were used in replicates 1, 2, and 5, respectively.

Sample condition was significantly different at the 95 percent level of probability. For every treatment, the indicated moisture level was higher for the frozen samples than the fresh samples. Those differences ranged from a high of 1.10 percent for treatment 2 to a low of 0.14 percent for treatment 11. Even though the frozen samples were significantly different, they only averaged 0.55 percent higher than the fresh samples. This would indicate that there is potential to use frozen samples. A large part of the differences could be attributed to treatment effects since the frozen samples tended to burn more

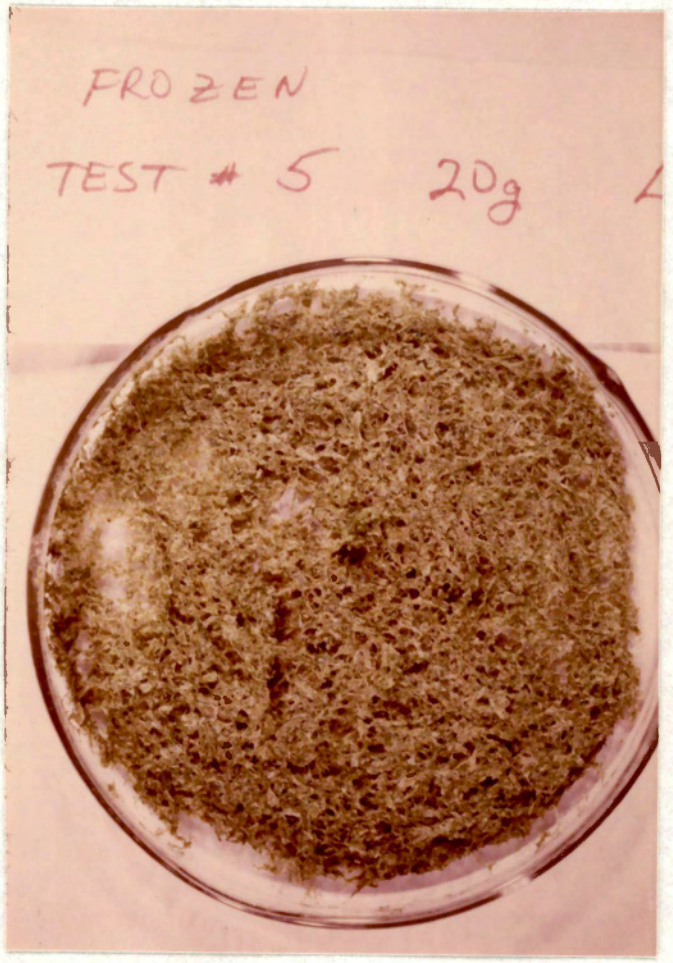


Figure 4. Sample condition of treatment 13 after drying 20 grams at oven setting 4 for 24 minutes.

readily than the fresh samples. Physiological differences caused by cell breakdown from freezing the beans could have accounted for the difference.

IV. PHASE FOUR (VACUUM, CONVECTION, AND MICROWAVE)

The indicated moisture content (wet basis) was evaluated as a function of oven pressure. The indicated moisture content was also compared among microwave, vacuum, and convection oven drying.

Vacuum and Convection Oven Drying

Before individual treatment effects (oven pressure) could be analyzed, the test effects and sample by treatment interactions had to be evaluated. Both the test effects and sample by treatment interactions were non-significant at the 95 percent level of probability. This indicated that each treatment (oven pressure) was affecting samples in the same manner. Thus, eliminating test effects and sample by treatment interactions, a less sensitive model could be used to analyze treatment effects.

Oven pressure (absolute pressures of 760, 660, 360, and 60 mm of mercury) was not a significant factor at the 95 percent level of probability. Oven pressure (absolute pressures of 660 and 60 mm of mercury) was also found to be non-significant in phase one. Wilhelm (1979) found that absolute pressures of 735, 710, and 660 mm of mercury were non-significant. An analysis of variance and orthogonal contracts of the treatments are given in Table 11.

Table 11. Vacuum and Convection Oven Drying Analyses and Orthogonal Contrasts for Phase Four

Source	DF	Type I SS	F Value	PR > F	DF	Type III SS	F Value	PR > F
Treatment	4	0.00023922	0.49	0.7408	4	0.00018124	0.37	0.8246
Test	11	0.00063304	0.47	0.8977	11	0.00063304	0.47	0.8977

Contrast	DF	SS	F Value	PR > F
660 vs 360	1	0.00007822	0.65	0.4296
660 vs 60	1	0.00000008	0.00	0.9803
660 vs 760	1	0.00000816	0.07	0.7980
660 vs Convection	1	0.00002476	0.20	0.6562
360 vs 60	1	0.00008379	0.69	0.4156
360 vs Convection	1	0.00003478	0.29	0.5981
60 vs 760	1	0.00000666	0.05	0.8171
60 vs Convection	1	0.00002823	0.23	0.6347
Pres Vacuum vs 760	1	0.00004914	0.41	0.5316
Pres Vacuum vs Convec	1	0.00000428	0.04	0.8528
Vacuum vs Convection	1	0.00002364	0.19	0.6635

Source	DF	Sum of Squares	Mean Square	F Value	PR > F	R-Square	C.V.
Model	15	0.00087226	0.00005815	0.48	0.9245	0.264530	1.2705
Error	20	0.00242513	0.00012126	Std Dev		M Mean	
Corrected total	35	0.00329739		0.01101165		0.86670226	

Vacuum versus convection oven drying was not significant at the 95 percent level of probability. Table 11 gives the analysis and contrasts for vacuum versus convection oven drying. In phase one, vacuum versus convection oven drying was significant. Since the temperature in oven (CQ) could be controlled much more closely than in oven (CL), the exclusion of oven (CL) could explain part of the non-significance in phase four.

Microwave Oven Drying

Ten-gram samples were dried at setting 6 (treatment 6) for the microwave oven test. Samples were obtained from the same sources used in the vacuum and convection oven section of phase four. The samples used were frozen even though sample condition was found to be significant in phase three. Frozen samples had to be used because fresh samples could not be held for the period of testing. The mean indicated moisture content for all four samples was 85.90 percent.

Comparison of Microwave Versus Vacuum and Convection Oven Drying

Since vacuum and convection oven drying differences were non-significant, the indicated moisture content obtained by the microwave oven was compared to an average of both the vacuum and convection ovens. This was done using the Students'-T distribution. The hypothesis was that the means of the indicated moisture content obtained by the three ovens were equal. Mathematically, the hypothesis, Ho, is

$$Ho: u_1 = u_2$$

where

u_1 = population mean for the vacuum and convection ovens combined, and

u_2 = population mean for the microwave oven.

For the comparison of two means, the value for Students'-T may be calculated from the equation

$$T = \frac{\bar{X}_1 - \bar{X}_2 - (u_1 - u_2)}{\left(S_1\left(\frac{1}{N_1}\right) + S_2\left(\frac{1}{N_2}\right)\right)}$$

where

T = Students'-T value,

$\bar{X}_1$ = vacuum and convection oven sample mean,

$\bar{X}_2$ = microwave oven sample mean,

N_1 = number of vacuum and convection oven observations,

N_2 = number of microwave oven observations,

S_1 = vacuum and convection oven standard deviation, and

S_2 = microwave oven standard deviation.

Since the population means were hypothesized to be equal, their difference was zero and they were not involved in the calculation. The tabular T values were selected at the 95 percent level of probability for a two-tailed test (0.025 probability that a greater T magnitude exists). The calculated T, which was 2.44, was greater than the tabular T (2.00). Therefore, the hypothesis failed and microwave oven drying was declared different from vacuum and convection oven drying. Table 12 lists the mean indicated moisture levels for each sample for microwave, convection, and vacuum oven drying. Even though the moisture contents given were statistically different, the variations were less

than 1 percent. A 1 percent error in a determination could be sustained for some applications when one considers the reduced drying time (from 24 hours to 12 minutes) offered by the microwave method.

Table 12. Average Moisture Levels for Samples Used in the Comparison of Microwave and Oven Drying

Sample	Indicated Moisture Content (percent)	
	Microwave	Vacuum and Convection
1	85.01	85.53
2	85.35	85.12
3	89.17	89.73
4	84.59	86.30

CHAPTER V

SUMMARY AND CONCLUSIONS

I. SUMMARY

Presently, there is no single method being used by researchers as a standard procedure for moisture determination in snap beans, a high moisture level vegetable. Past research has dealt mainly with dried fruits and canned and frozen vegetables. No record was found of research using microwave energy to determine moisture rapidly in snap beans. A review of literature which described the use of microwave energy for determining moisture in other products prompted the selection of microwave energy to rapidly determine moisture in snap beans.

A study was conducted to evaluate the performance of microwave energy to determine moisture content rapidly and to identify a standard oven drying procedure for determining moisture in snap beans.

The experiment was divided into four phases. In phase one, frozen bean samples were dried in two convection ovens and two vacuum ovens to evaluate the effects of drying temperature and oven pressure. The convection ovens were operated at temperatures of 65 and 100°C, while the vacuum ovens were operated at 65 and 100°C with absolute pressures of 660 and 60 mm of mercury. In phase two, fresh and frozen bean samples were dried in two vacuum ovens to evaluate the effect of sample condition (fresh vs. frozen). The vacuum ovens were operated at 100°C and an absolute pressure of 60 mm of mercury. In phase three,

fresh and frozen samples were dried in a microwave oven to evaluate the effects of sample condition and treatment combinations of sample size and power setting. Sample sizes used were 5, 10, 15, and 20 grams and power settings were 4, 6, 8, and 10 (full power). In phase four, oven pressure along with a comparison of convection, vacuum and microwave oven drying were evaluated. The pressure levels in the vacuum ovens were 760, 660, 360, and 60 mm of mercury (101, 88, 48, and 8 kPa). Both the vacuum and convection ovens were operated at 100°C. The microwave oven dried 10-gram samples at power setting 6 (treatment 6), which was found to be the most reliable treatment combination in phase three.

II. CONCLUSIONS

Convection and Vacuum Oven Drying

Of the parameters studied in this test, none was found to be significant in the ranges used. However, indicated moisture content was more sensitive to temperature than any other factor. Thus, convection ovens operating at 100°C and vacuum ovens operating at 100°C with an absolute pressure between 760 and 60 mm of mercury (101 and 8 kPa) gave the same indicated moisture content. The drying process required 24 hours. Either fresh or frozen samples can be used.

Microwave Oven Drying

Of the parameters studied in this test, three were found to be significant. Sample size combined with oven power setting (energy level) had a major effect upon the indicated moisture content. The

larger sample sizes lengthened drying times. Higher power settings produced higher indicated moisture contents. Sample condition (fresh vs. frozen) was significant with the frozen samples giving higher indicated moisture contents. A thorough grinding of the bean samples with a food processor satisfactorily prepared both fresh and frozen samples.

Microwave oven drying was significantly different from convection and vacuum oven drying at the 95 percent level of probability. However, for each sample tested these differences were less than 1 percent. Thus, microwave samples dried for 12 minutes gave indicated moisture contents that closely approached indicated moisture contents given by vacuum and convection oven samples dried in 24 hours.

Further testing is needed to compare microwave drying with conventional oven drying. Microwave energy has much potential for moisture determination because of its ability to reduce drying time.

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