

Sanitizing Spice Seeds

by

Ayad Abdalla Ayad

B.SC., Alexandria University, 1965

A Master's Thesis

submitted in partial fulfillment of the

requirements for the degree

Master of Science

Department of Grain Science and Industry

KANSAS STATE UNIVERSITY

Manhattan, Kansas

1975

Approved by:



Major Professor

LD
2668
T4
1975
A93
C.2
Document

TABLE OF CONTENTS

	Page
INTRODUCTION	1
REVIEW OF LITERATURE	3
MATERIALS AND METHODS	16
RESULTS AND DISCUSSION	22
Cleaning of Spices	22
Total Count of Microorganisms	23
Chemical Treatments	25
Volatile Oil Content	33
Insect Contamination	36
SUMMARY	37
ACKNOWLEDGMENTS	38
LITERATURE CITED	39

INTRODUCTION

Spices are any of various aromatic vegetable products or condiments such as pepper, cumin, celery and others used in cookery to season food, and to enrich or alter the quality of a food by adding zest or pungency. They also have a preserving effect (1). Their chemical compositions are of great interest to the spice manufacturer. The commercial value of crude spices, in general, varies according to the percentage of volatile oil content (2). Pepper alone normally accounts for over one fourth of the total world trade in spices. At the present time, the volume of international trade in spices amounts to about \$170 million a year. Generally speaking, spices are far more important to the economies of the producing countries than to those of the consuming countries. Demands for most spices is fairly constant but supply may vary. Spice prices, therefore, are very unstable and may fluctuate considerably from year to year. The average import price in the U.S.A. during 1967-1968 exceeded \$100 a pound. In the United States there has been a growing interest in spices during the past twenty years. This country is, by far, the world's largest importer of spices, importing in 1970 over 250 million pounds. The European economic community (West Germany, France, Italy, Netherlands, Belgium and Luxembourg) now imports annually about \$35 million worth of spices; the Soviet Union, \$12 million and the United Kingdom, \$7 million (1).

Safe storage of spice seeds is an important biological problem. Microbes require certain ranges of temperature, moisture and food supply

to survive and grow. Microflora on spices during storage often cause heating. Microorganisms found on the spice seeds produce heat just as other living organisms that respire. Stopping growth of microorganisms in spices is the most important factor related to safe storage. Many methods have been used to remove the microorganisms and to prevent or minimize deterioration of high-moisture, stored spices. One method has involved the addition of propionic and acetic acid and ethylene oxide to inhibit microorganism growth.

This investigation deals with means to clean and sanitize various common spices. By cleaning is meant the removal of particles smaller and larger than spices. By sanitizing is meant the removal of microorganisms normally found on the seed coat of spices.

REVIEW OF LITERATURE

The first authentic record of the use of spices and herbs may have been during the pyramid age in Egypt, approximately 2600 to 2100 B.C. The earliest mention in the Bible of an aromatic substance is in Genesis 2:12 in a reference to the land of Havilah (1). King Solomon received tribute in spices from the Queen of Sheba who brought him spices as a tribute to his great wisdom. Spices then were worth more than their weight in gold (3).

A wealth of information concerning ancient herbs and spices was discovered by the German Egypt-ologist George Berbs in a remarkable medical document dated about 1550 B.C. This comprehensive study, reported by him in 1874, is now known as the Ebers papyrus. It documented that Egyptians used aromatic spices in medicine, cosmetics, ointments, perfumes, and aromatic oils, cooking, fumigating and embalming (1).

In 2450 B.C. Pates of Lagash, when preparing to build a temple, purified the city by building a fire of aromatic woods, probably the first use of spices for fumigation. Although spices have been known for many centuries, the Chinese seem to have been the first to organize a substantial spice business (1).

During the first three centuries of the Christian era spices were very valuable articles of exchange and trade. Rome did an active business with pepper from China. The center of the pepper trade was Calicoda, in what is now India. When the Roman Empire fell, its spice trade died. By 8 A.D. the Arabian Empire extended from Spain to Western China. Food in Europe was neither good nor palatable until spices were

rediscovered. It was found that a dash of pepper mixed with the coarsest dishes would change their taste period. Demand for spices soon made them the most precious of all commodities (3). Through history, the country that controlled the spice trade was the richest and most powerful in the world. Fortunately, the aromatic flavors are not so costly today (3).

Safe storage of spices is an important biological problem. The average loss caused by microorganisms during storage is large, costing millions of dollars annually. Growth of microorganisms in most spices is perhaps the factor most related to the safe storage. Microbes require certain ranges of temperature, moisture and food supply in which to grow.

Fungus spores can germinate at a low critical moisture level of 14%. Some species of fungi, such as the Aspergillus glaucus, can be grown at lower moisture than other species. Under these conditions more moisture will accumulate due to respiratory activity and other species of fungi may develop (4). More recently, it has been established that growth of fungi causes excessive heat (4-5). Deterioration in quality can also be caused by the fungal enzymes. These enzymes attack the carbohydrates, fats and proteins causing high acidity and deterioration of protein and starch (4).

Many methods have been introduced to control microbes, sterilization is the process of destroying all living forms of life. Disinfection is the process of destroying all vegetative forms of microbes, but not necessarily spore forms. Sanitization refers to destruction of microbes so as to leave a microbe population within limits as established by public health requirements (6). In actual practice the difference between sterilization, disinfection and sanitization is simply one of severity of action of the sanitizing method or agent used. Some methods

used are primarily through their physical action. Application of heat was one of the first methods. Heat is one of the most reliable and readily controlled sterilizing methods available (7). Effectiveness of heat application depends on several major factors; type and number of microbes, physiological state of microbes, properties of the food material and length and degree of exposure. The pH of moisture of food media are probably the most important food characteristic (8). Term and exposure to heat are extremely important. Also dry heat is less effective than moist heat (9). Hot air at 180°F for 20 minutes is equivalent to water at 170°F for 2 minutes (7). In dry heat microbes may be destroyed by oxidation, while in moist heat death may occur because the protein of the microbes coagulates on inactivation of enzymes (10-11).

Baqir (12) has worked with a grain sanitizer in which whole kernel grain is passed through a high temperature flame to kill microbes, mainly molds. His work has verified damp grain may be safely stored when the grain is passed through an oxidizing flame of high temperature.

Moist heat, as in an autoclave, is quite effective depending upon time and peak temperature. No living organism can survive ten minutes of direct exposure to steam at 121°C.

Dessication is another type of microbial control. In this case, most microbes are not exterminated, but they are capable of new growth once sufficient moisture is present.

Another major method in microbial control is refrigeration. The effect of cold on microorganisms is not well known or described (13). As food is frozen, the cold slows all biological processes, but complete animation is never attained.

Another physical method is the use of filters. The smallest bacteria are 0.15 micron in diameter by 0.3 micron in length, with the average food bacteria being 0.5-2.5 micron in diameter by 2-10 micron in length. Yeast and molds are much larger (13). Filtration is the only effective way of physically removing microbes from fluids and a fairly effective way of removing microbes from air and liquid.

Chemical Methods

Effectiveness of a chemical as a sanitizing agent is affected by kind and concentration of the chemical, time of exposure, temperature of substrate, type of substrate and, type and number of organisms. The effect of an increase in germicidal effect of a chemical with a slight temperature increase has been documented (7). Also, an addition of surfactant to a chemical may greatly increase the germicidal effect of many antimicrobial chemicals (7). The pH of the substrate has a pronounced effect both on the action of the chemical and also on the control of microbes. Molds and yeast are quite acid tolerant, while bacteria vary greatly in response (14). Chemical compounds inhibit growth of microbes in several main ways; in activation of vital metabolite, denaturation of cellular protein or direct physical damage (13). A given compound may be effective against certain organisms and only under certain conditions. Some work has been done using acid sprays to disinfect large open spaces (10). Stevenson (15) also treated damp grains with propionic acid and a mixture of acetic and propionic acids. By the strong vinegar like vapors and the low pH of acetic and propionic acids, growth of microorganisms on the grain were inhibited. Moist grain can be stored without deterioration if treated with acids such as

propionic. It was also stated that while propionic acid has more effect as a fungicide, acetic acid is better than propionic as a bactericide (15).

Gaseous antimicrobial agents have definite applications in public and medical health but have not had large applications to food processing. Some gasses which have been used for sanitizing includes chlorine, ethylene oxide, formal dehyde, ozone, propiolactone and sulfur dioxide (10-11). Ethylene oxide has good penetration power, a wide range of antimicrobial effect and leaves no toxic residue in food but is slow acting and potentially explosive (16). However, its actions and limitations have been well investigated and it is very useful if these limits are observed (17).

It appears that the effect of ethylene oxide on grains is similar to that observed by Denny (1924) on lemons. The possibility has indicated that the treatment of damp grains with ethylene oxide might materially increase its keeping qualities in storage by controlling the growth of microorganisms. It was found that when damp wheat was exposed to ethylene oxide, its keeping quality was decidedly improved as compared with untreated wheat. One effect of the ethylene oxide treatment was an immediate increase in CO_2 output of the grain. It does not seem likely that this increase could have been due to the development of microorganisms, but rather to astimulation of metabolism in the seed (18-19).

Cigarette Beetle (*Lasioderma Serricorne* F.)

The cigarette beetle is primarily a pest of stored tobacco but it occasionally attacks stored spices. It is cosmopolitan by nature, being widely distributed over the world. It can breed in a number of foods,

including pepper and seed products. The adult beetle is light brown in color with head bent downward giving it a humpbacked appearance. It is small and the eggs are laid among or on the larval food and hatches in one to two weeks at summer temperatures. The entire life cycle is passed in about 50 days (20).

Spices and herbs are classified in many different ways but some of the most frequently used terms are as follows: Aromatic spices are those that have an especially pronounced fragrant flavor, such as cumin and fennel. Stimulating spices include those that have an especially zesty, pungent flavor such as pepper. Sweet herbs are those plants whose leaves, stems or roots have an aroma flavor that makes them adaptable to cookery. Thyme and fennel are among the sweet herbs.

Celery Seed (*Apium Graveolens* L.)

Celery seed is the dried fruit of a biennial herb of the parsley family, native to Southern Europe and Northern Africa (1). It is cultivated for its fruits in the U.S.A., most of the celery fruit imported into the U.S.A. comes from India and France (3-21). The Romans and Greeks grew celery for its medicinal qualities rather than as food (1). The fruits are collected when fully ripe (21). The seeds are light brown in color and they are small in size, rarely exceeding 1/16" in length. Celery seed flavor is almost identical with that of the celery vegetable, warm and slightly bitter (3).

Celery fruit contains volatile oils, fixed oil, resin, protein, cellulose, calcium oxalate and mineral elements. The principal constituent of celery fruit is its volatile oil containing about 2 to 3%. The characteristic odor is due to two oxygenated compounds present in the oil

namely sedanolide and sedanonic acid anhydride. The fixed oil content of celery is about 16%, the fatty acid consists largely of petroselinic, oleic and palmitic acid (22). Celery seed oil is used to flavor food products and in liqueurs, perfumes and soaps.

In California, the celery seedlings usually are started in the summer and transplanted in the fall to the field. Celery seed production in the U.S.A. is wholly inadequate for domestic needs and much is imported into this country. Most of the celery grown in the U.S.A. is produced for the vegetable market (1).

Cumin Seed (Cumin Cyminum L.)

Cumin is a small annual herb of the parsley family, believed to be a native of upper Egypt, Turkistan, and the Eastern Mediterranean region (1). U.S.A. imports most of the cumin seed from Iran and Morocco (3). It is not grown in the United States on a commercial scale (1). The dried fruit, commonly called cumin seed, consists of united or separate carpels. Cumin seed has a distinctive heavy, strong odor and a warm, aromatic taste (21). In 1968, the average price of cumin seed from Iran imported into the United States was 14 cents per pound. In indigenous medicine in India, cumin seeds have traditionally been employed as a stimulant, carminative and useful in diarrhea and colic (1). Cumin fruit contains volatile oil, fixed oil, protein, cellulose, sugar, pentosans, calcium oxalate and mineral elements. Cumin fruit contains from about 2.5 to 4.5% of volatile oil, the chief constituent of the oil is cumaldehyde. The fixed oil content of cumin is approximately 10% (22).

Cumin is another spice which historically predates Biblical times. In the twenty-third chapter of Matthew, Jesus said: "Ye pay

tithe of mint and anise and cumin (3). The essential oil is used in making liqueurs and also to a small extent in perfumes (23).

Fennel Seed (Foeniculum Vulgare Mill)

The dried fruit is a perennial herb of the parsley family and is a European native. The United States imports most of the fennel seed from India and Romania. The seed has an agreeable odor and an aromatic, sweet taste (3). The flavor of fennel varies greatly according to the type (24). The seed of fennel is a useful spice with a flavor something like anise but not so sweet. It had a great reputation in medicine long before the plant was used as a culinary herb. In fact, it is a good digestive and also an aid in weight control (24).

Fennel fruit contains volatile oils, fixed oil, protein, cellulose, sugar, pentosans, calcium oxalate and mineral elements. Fennel fruit contains from about 3 to 4% of volatile oils. The chief constituents of the oil are nethol and fenchone. The fatty acid consists largely of petro selinic, oleic, linoleic and palamitic acids (22).

All of the fennels are easy to grow as annuals but require a long growing season and for this reason most of the production in the U.S.A. is in California (25).

Black Pepper (Piper Nigrum L.)

Pepper is the world's most important spice (1). Although from very ancient times pepper has been consumed through the world, but its production has always been restricted to a comparatively small area in the Far East. Originally pepper was moved overland by Caravan through India and Persia, or by water through the Persian Gulf or the Red Sea. Pepper was moved up from the Red Sea and then taken by Caravan to the

Nile. Venice and Genoa became secondary points of distribution to England and Northern Europe. The pepper was said to be worth its weight in gold and was so valuable that it was used as money. In the early days of the U. S., the American clipper ship played a very important role in the transportation of pepper. During the first half of the 19th Century a large proportion of the pepper was carried by the clipper ships, handled by Salem merchants and distributed to other countries. In recent years, there has been a rapid shift in the relative importance of pepper market. In the early thirties, the control of pepper shifted to London and in the late thirties, to New York City. Today, a dominant role in the pepper trade does not have the political, economic or social significance that it had 500 years ago (3).

Pepper is the dried fruit of Piper Nigrum L. belonging to the Piperaceae or pepper family. The fruit, commonly called berries or peppercorns, are small nearly spherical and dull green in color when immature, turning yellowish and finally red as they ripen. In Asia it is the practice to steep the red berries in boiling water for about ten minutes and spread on mats to dry in the sun. Drying is carried out as rapidly as possible to prevent or minimize mold growth, and with good sun, the process is complete in 3-4 days. The hot water treatment makes the skin turn black in an hour. The end product is dried black pepper (21).

Pepper contains volatile oils, fixed oils, resins, alkaloids, proteins, cellulose, pentosans, starch and mineral elements. Pepper contains from 1 to 3% volatile oil. Essential oils are volatile oils that may be vaporized at room temperature (22). Black pepper has a characteristic penetrating, aromatic odor, and a hot, biting very pungent taste (22). Much of the volatile oil and other properties that make

pepper valuable are in or near the outer husk of pepper. Pepper has a wide variety of uses in the food, drug and toilet industries. It is used to flavor and to preserve foods. Pepper aids in digestion since it stimulates the flow of digestive juices. Pepper is used by the medical profession but not as extensively now as in the middle ages (3).

Enormous quantities of pepper are consumed annually. It is estimated that the world's consumption amounts to approximately 60,000 tons yearly. Of this, the United States uses about 15,000 tons a year while the balance is consumed in Europe and the Far East, India being the largest buyer (2). No commodity has had a more romantic history than pepper.

Thyme (Thymus Vulgaris L.)

Thyme is a diminutive perennial herb of the mint family native to the Mediterranean region and Asia Minor. It is grown in California although most thyme consumed in the U.S.A. is imported from France. The herbage is harvested when the plants are in bloom, the flowering tops are cut off together with several inches of the tender, leafy stem. This herbage is shade-dried to preserve the grayish-green color (1). The dried leaves have a fragrant, aromatic odor particularly when crushed and an aromatic, warm pungent taste (21). Thyme is strongly aromatic and contains more or less volatile oil called thymol which is a disinfectant. Aromatic properties of thyme is well liked in seasonings and it is used in the medical trade (2).

Oregano (Origanum Vulgare L.)

Oregano, derived from the Greek word means "Joy of the Mountain" and was popular in Ancient Egypt and Greece as a condimental flavoring for wine (1). Oregano is indigenous to the Mediterranean region and

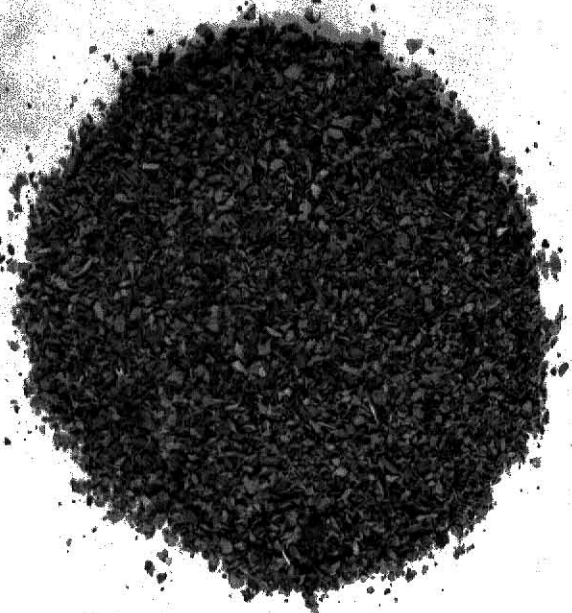
Western Asia. It belongs to the Labiae or mint family (21). Oregano contains volatile oils, fixed oils, protein, cellulose pigment and mineral elements (22).

Oregano's flavor is strong and aromatic with a pleasantly bitter under-tone. Its flavor is similar to majoram (3). Oregano has been used by physicians as a stimulant, nerve tonic and cures for asthma coughs, indigestion, rheumatism, toothaches, headaches, spider bites and coronary conditions. On steam distillation, the dried leaves yield 0.2 to 0.8% volatile oils. The Spanish oil known as Spanish wild marjoram oil, is utilized widely in U.S.A. as a flavoring in the preparation of food. In the U.S.A., oregano is grown commercially for the production of the dried herb. Oregano is imported to the U.S.A. from Greece, Mexico, France, Italy and Turkey (1).

Plates 1 & 2 illustrate the whole spices used in the study.

LAMPONG BLACK PEPPER**INDIA CELERY SEED****IRANIAN CUMIN SEED****FENNEL**

MEXICO OREGANO



SPANISH THYME



MATERIALS AND METHODS

The following spices were obtained from Spicecraft Inc., St. Louis, Missouri: India celery seed, Iranian cumin seed, fennel seed, Lampong black pepper, Spanish thyme and Mexican oregano. The Carter dockage tester, with appropriate sieves and air aspirator, was used to mechanically clean the spices. In black pepper the screen used in the Carter dockage tester had a sieve of 8/64" opening. The air control was set at 3.5 lbs/minute and the feed control at 4.5 lbs/minute. Samples of black pepper were examined for hidden infestation by use of the Picker X-ray apparatus. The sample of black pepper was treated with flash heat (two pounds in each treatment) according to Table 1.

Fennel, cumin and celery seeds were also cleaned on the Carter dockage tester and the aspirator. The samples of spices were examined visually for any insect infestation, and were treated with flash heat according to Table 1.

Plate count method was used to determine the number of microorganisms in spice seeds. Eleven gram samples were drawn from each treatment to make the total count of microorganism (molds, bacteria and yeast) colonies per gram. Potato dextrose agar (PDA), after acidifying with tartaric acid, was used for growth of molds. Nutrient agar (NA) was used for bacteria and yeast growth. A series of dilutions were made from each treatment as follows: 1:10, 1:100, 1:1000, 1:10,000, 1:100,000. Triplicate agar plates per each dilution were incubated at 30°C for five days before counting the microorganism colonies. If the counts varied greatly, the two plates with close agreement on counts were chosen. If

Table 1.

Flash Heat Treatment of Indicated Spice Seeds
(Two samples of each; one treated once; one treated twice)

Control	Black Pepper	Fennel	Cumin	Celery	Degrees C
		A 2/	A	A	400
	A	A	A	A	500
	A	A	A	A	600
	A	A	A	B 3/	700
	A				800
	A				850

1- Passed through the flash heat in 0.1 second.

2- A indicates two treatments (one treated once; one treated twice).

3- B indicates one treatment.

the counts were between 30 and 300 colonies, the data were accepted and calculated according to the number per gram according to the dilution proceeding this count. If the count was less than 29 and more than 300 colonies per plate, plates having the 30 to 300 count of different dilutions were used. Total count of molds, bacteria and yeast was performed just after treatment. Bosh-Lamp colony counter was used for this purpose.

An instrument was designed and built to treat spice seed samples with flash heat at different temperatures. This instrument consisted of a 60" X 5" steel tube with six baffle plates located alternately inside the tube to regulate flow of spices. Two gas burners were installed inside the tube at 40" and 45" from the top of the gate to which a hopper was attached. These burners were connected to gas-air mixing valves from outside. Compressed air and natural gas were used as a source of fuel. A 9" long hopper with slide and ventilation gates was attached to the top of the tube, while a metallic 10" X 8" X 6" receiving box was attached at the bottom of the tube to receive the spices after treatment. To measure the temperature of flash heat, a pyrometer was inserted just above the burners. The instrument was held vertically by a triangular iron stand. Treated spice samples were passed quickly through flash heat into the receiving box. The average time for seed to pass through the intense heat (500-700°C) was 0.1 second. Since the apparatus had a gate to regulate flow of the seed, the rate of passage of spice seed was regulated at 4 pounds/minute. This instrument is illustrated in Plate 3.

The Spanish thyme and Mexican oregano was mechanically cleaned by using an aspirator with a certain amount of air. Samples of the thyme and oregano were examined visually for any insect infestation. The

samples of thyme and oregano were treated with ethylene oxide for 20, 15, 10 and 5 minutes injected into an evacuated steel tank. The air tank was evacuated from air for two hours by using an evacuating air pump.

Effect of Flash Heat on Insect Infestation

Samples of black pepper, cumin, fennel and celery originally cleaned from infestation were artificially invaded with cigarette beetle. The samples were incubated at 70°F temperature and 75% R.H. for 55 days. The sample was treated with flash heat to determine the effect of the flash heat on the insect population. Black pepper was treated once at 800°C, cumin, fennel and celery seed were treated once at 600°C and the samples incubated at room temperature with 75% R.H. for ten days. Each spice was examined visually for growth of insects.

Plate 3 - Illustration of flash heat apparatus used for sanitizing spice seeds.

To the left of the tube can be seen a thermocouple leading to the pyrometer for recording temperature of flash heat. Just below the thermocouple are two natural gas burners attached to gas mixing valves. At the top of the tube is the hopper and regulating valve, while the receiving box is attached to the bottom of the tube. This box was located to receive the treated spice. The tube is equipped with baffle plates to spread the seed.

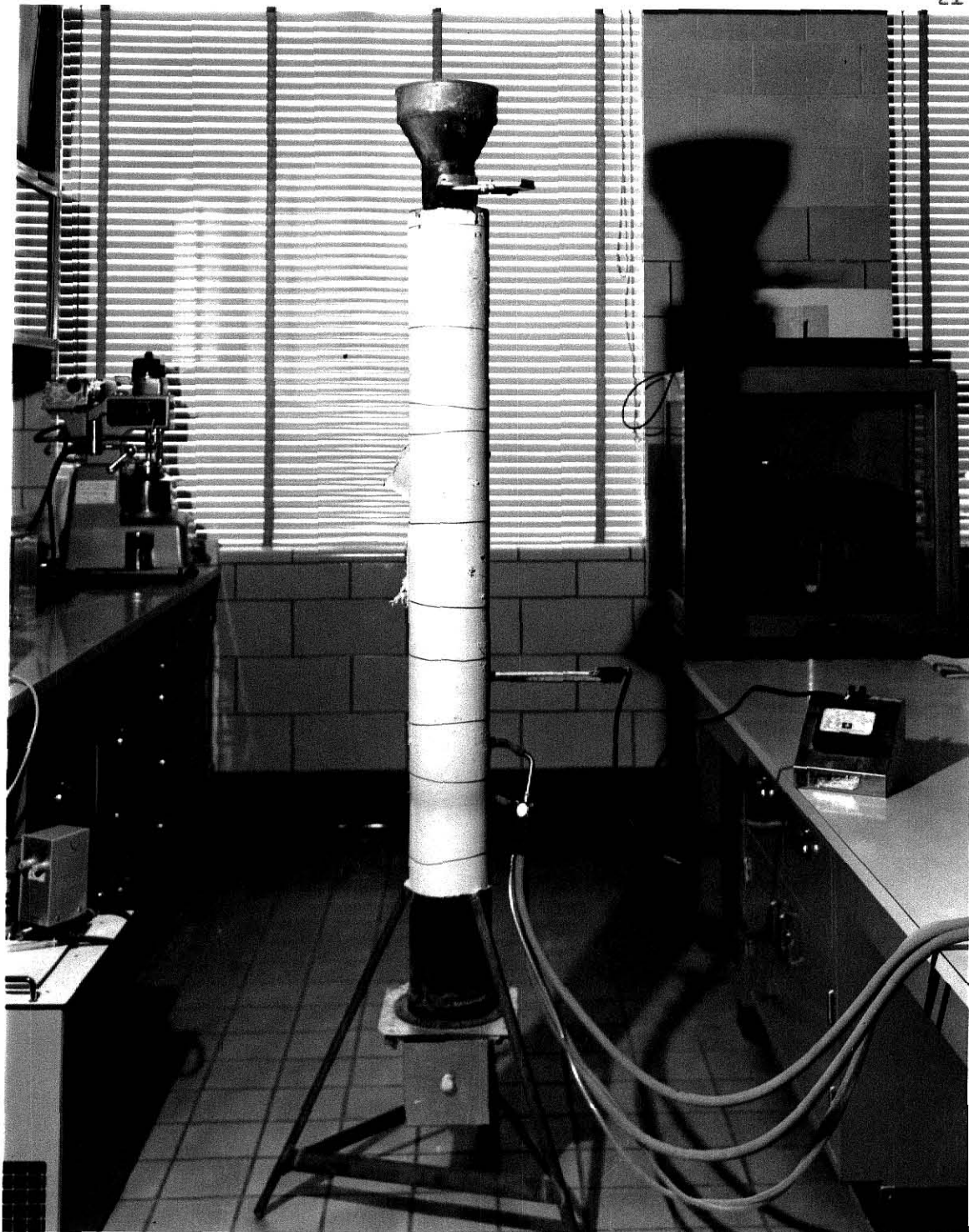


Plate 3

RESULTS AND DISCUSSION

Air cleaning of black pepper with a Carter dockage tester removed dust and chaff. This treatment removed 90 grams per 20.8 kg. sample. No live insects could be found in the air-cleaned sample of black pepper. In 1 pound, 2 black pepper were found with insects emergence holes. Examination of the black pepper by X-ray showed no evidence of the presence of insect larvae. It was concluded that the sample of black pepper was almost free of insects.

Air cleaning of fennel seeds with aspirator removed dust and chaff amounting to 428 grams per 21.5 kg. sample. No dead or live insects were found in the sample.

Air cleaning of cumin seed with aspirator removed dust and light cumin, 812 grams per 20 kg. sample. No live or dead insects were found in the sample.

Air cleaning of celery seeds with an aspirator removed dust and chaff, 1500 grams per 20.2 kg. sample. No live insects were found in the air-cleaned sample of celery seeds.

The microbial count can be best expressed in number of colonies per gram of spices. The microbial count of each of the spices before and after flash heat treatment are summarized in Table 2.

The data in Table 2 indicate that mold count on black pepper was steadily decreased as the temperature was increased or given two exposures to flash heat. The mold count reached an insignificant level when one flash heat treatment of 700°C was used. The bacteria + yeast appeared more resistant to destruction by flash heat treatment than the mold.

Table 2.

Microbial Counts in Spice Seed Samples Sanitized
by Exposure to Flash Heat as Indicated

Treatment Degrees C	Black Pepper		Fennel		Cumin		Celery	
	Average Molds Col./g	Average Bact + Y. Col./g	Average Molds Col./g	Average Bact + Y. Col./g	Average Molds Col./g	Average Bact + Y. Col./g	Average Molds Col./g	Average Bact + Y. Col./g
Control	320	7.1 (millions)	2500	27 (thousand)	1500	43,000	60	(thousand) 290
400			2500	20	300	20,000	Nil	109
400 T*			1000	15	200	20,000	0	74
500	550	7.1 ⁺	750	**	90	8,350	0	73
500 T	130	7.1 ⁺	500	15	Nil	3,100	0	61
600	180	7.1 ⁺	400	11	0	4,500	0	70
600 T	160	7.1 ⁺	0	9	0	200	0	9
700	Nil	7.1 ⁺	0	5	0	300	0	11
700 T	0	7.1 ⁺	0	5	0	300		
800	Nil	7.1						
800 T	0	4.3						
850	0	4.0						
850 T	0	2.01						

* Two 400°C Treatments

** Contaminated

The numbers were not significantly affected until a flash heat treatment of 800°C of two exposures were used. These facts are further illustrated by Figure I.

The treatment of fennel indicate that the mold was steadily reduced as the temperature was increased from 400 to 600°C with two exposures. Similarly the bacteria was present in much greater amount than the mold. These were steadily reduced as the temperature increased up to 700°C with one or two exposures. Since the surface of fennel seed was smoother and more readily exposed, it was evident that the flash heat treatment was more effective in removing the microorganisms than in black pepper. Figure II illustrates the destruction of the molds with increasing temperature and number of exposure. Figure III illustrates how treatment with flash heat reduced the number of bacteria + yeast in the fennel.

Increasing the temperature of the flash heat treatment or the number of exposures in cumin indicated that the number of microorganisms was significantly reduced. One exposure at 500°C significantly reduced the number of mold below treatment of 400 with two exposures. As the exposure was increased beyond 400°C and the number of exposures was increased to two the number of mold was significantly reduced. At 500°C with two passes, the number of molds were reduced to insignificant number. The number of bacteria + yeast was much greater than the number of molds but these too were steadily decreased as the temperature of the number of exposures increased. Treatment at 500°C with two exposures significantly reduced the bacteria over treatment at 400°C or 500°C with one exposure. Temperature above 700°C would seemly be unnecessary and not recommended because schorching of spice seed. The effect of flash heat treatment

on the number of mold of cumin is further illustrated in Figure IV, the number of bacteria + yeast of cumin is further illustrated in Figure V.

Treatment of the celery seed indicated that the low temperature of flash heat treatment at 400°C with two exposures reduced the number of molds to an insignificant number. Likewise, higher temperature reduced the mold count to zero. The bacteria on the celery seed again was much greater than the number of mold, but their number was steadily reduced as the temperature was increased particularly as the temperature reached 700°C . The effect of flash heat on the number of molds on celery is illustrated in Figure VI. The number of bacteria + yeast of celery is further illustrated in Figure VII.

Thyme

The mechanical cleaning of thyme removed 1.3 kg. of dirt and chaff from 20 kg. of material. This was equal to 6.5% of the total mass. No live insects were found in the air-cleaned sample of thyme. Since thyme is a leafy fine type of spice it could not be sanitized by flash heat treatment. Rather, it was necessary to treat with ethylene oxide to destroy the microorganisms.

The microbial count for the untreated sample was 45 thousand colonies of mold per gram, while the bacteria + yeast count was 6 million colonies per gram. After thyme was subjected to treatment with ethylene oxide for 5 minutes or longer, the mold and bacteria count was reduced to zero. These facts are further illustrated by Figure VIII.

Oregano

The mechanical cleaning of oregano removed 0.65 kg. of dirt and chaff from 10 kg. of material. This was equal to 6.5% found in the

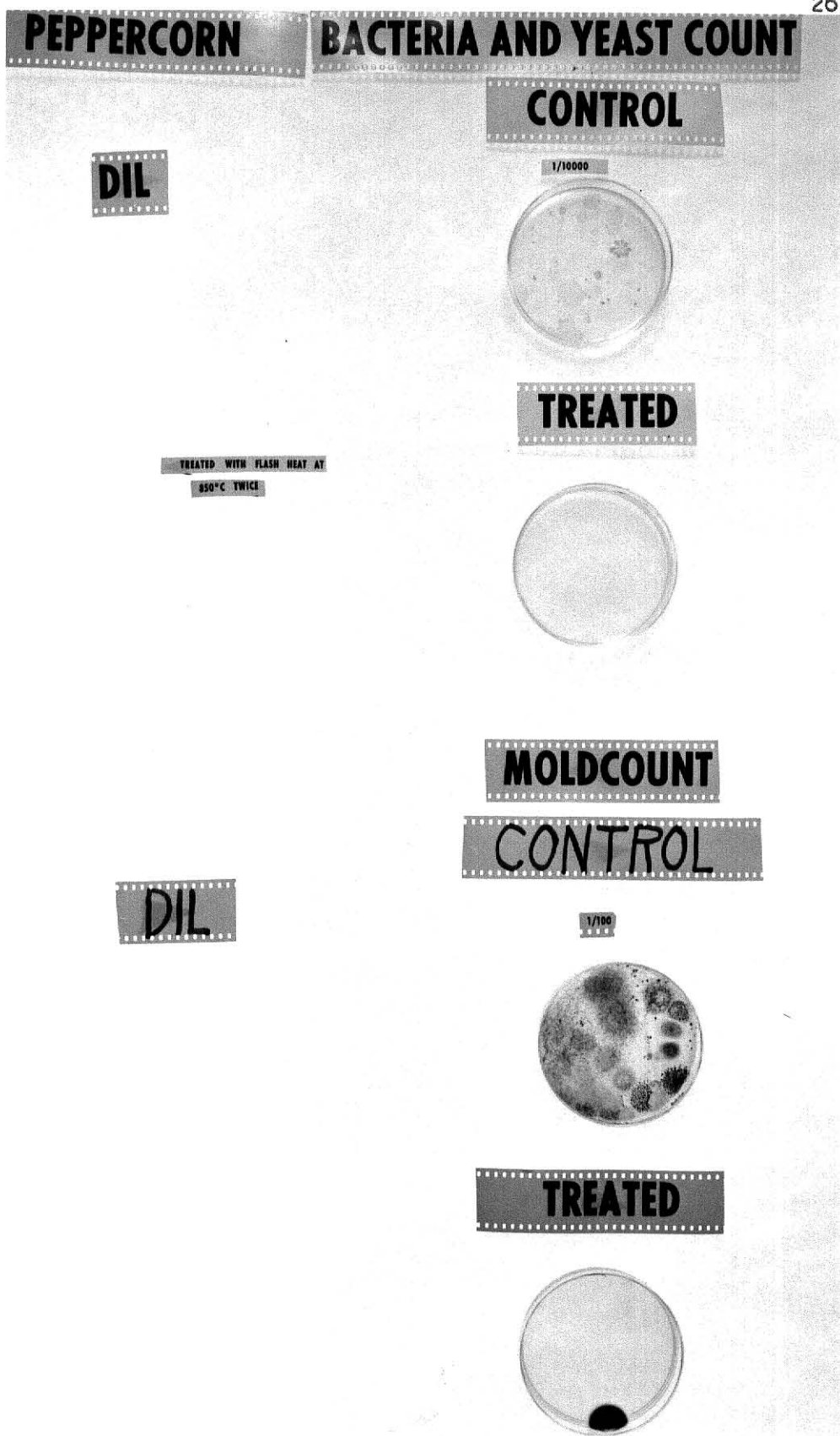


Figure I. The number of bacteria + yeast and mold before and after treatment with flash heat.

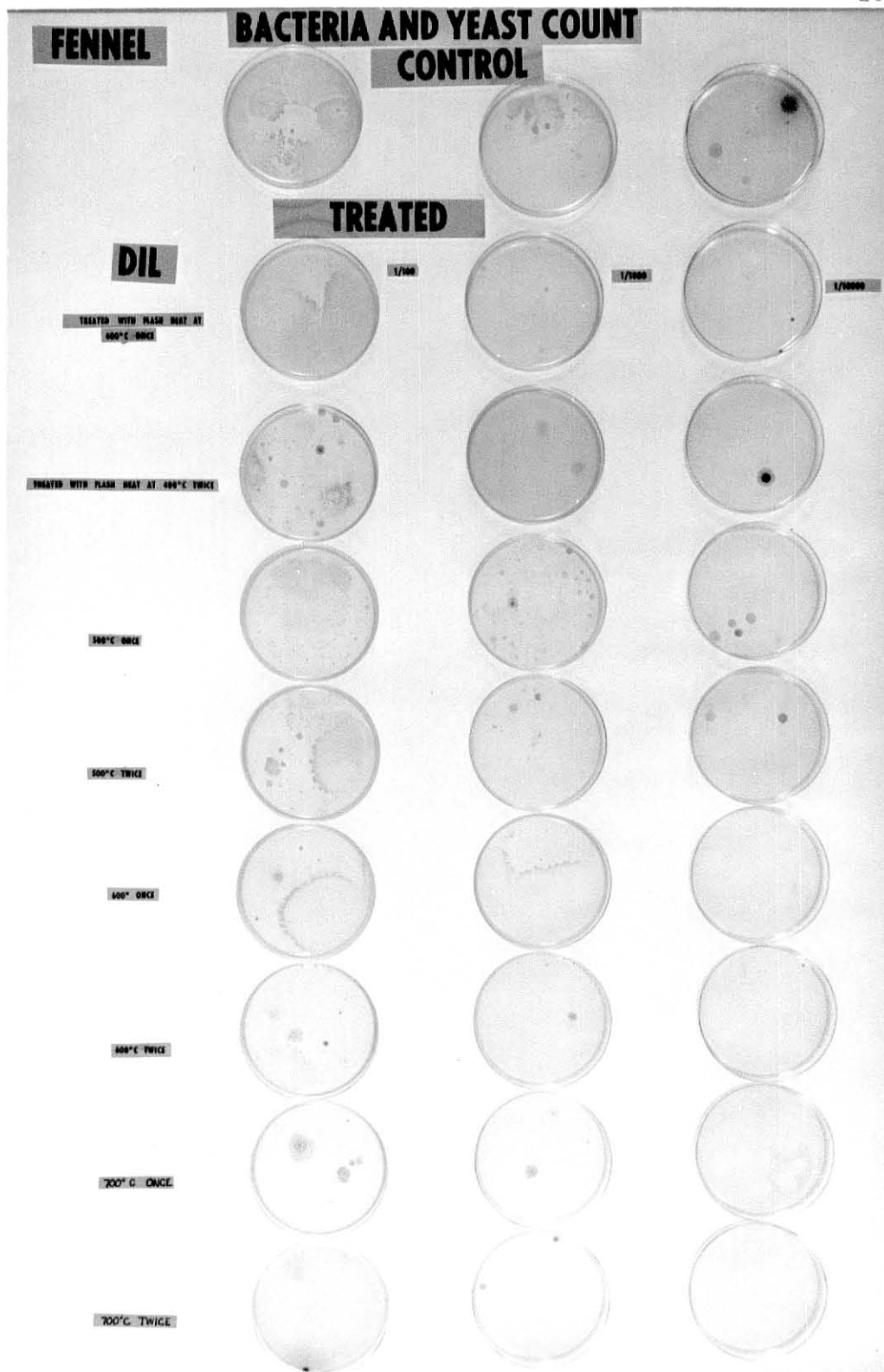


Figure III. The number of bacteria + yeast before and after treatment with flash heat.

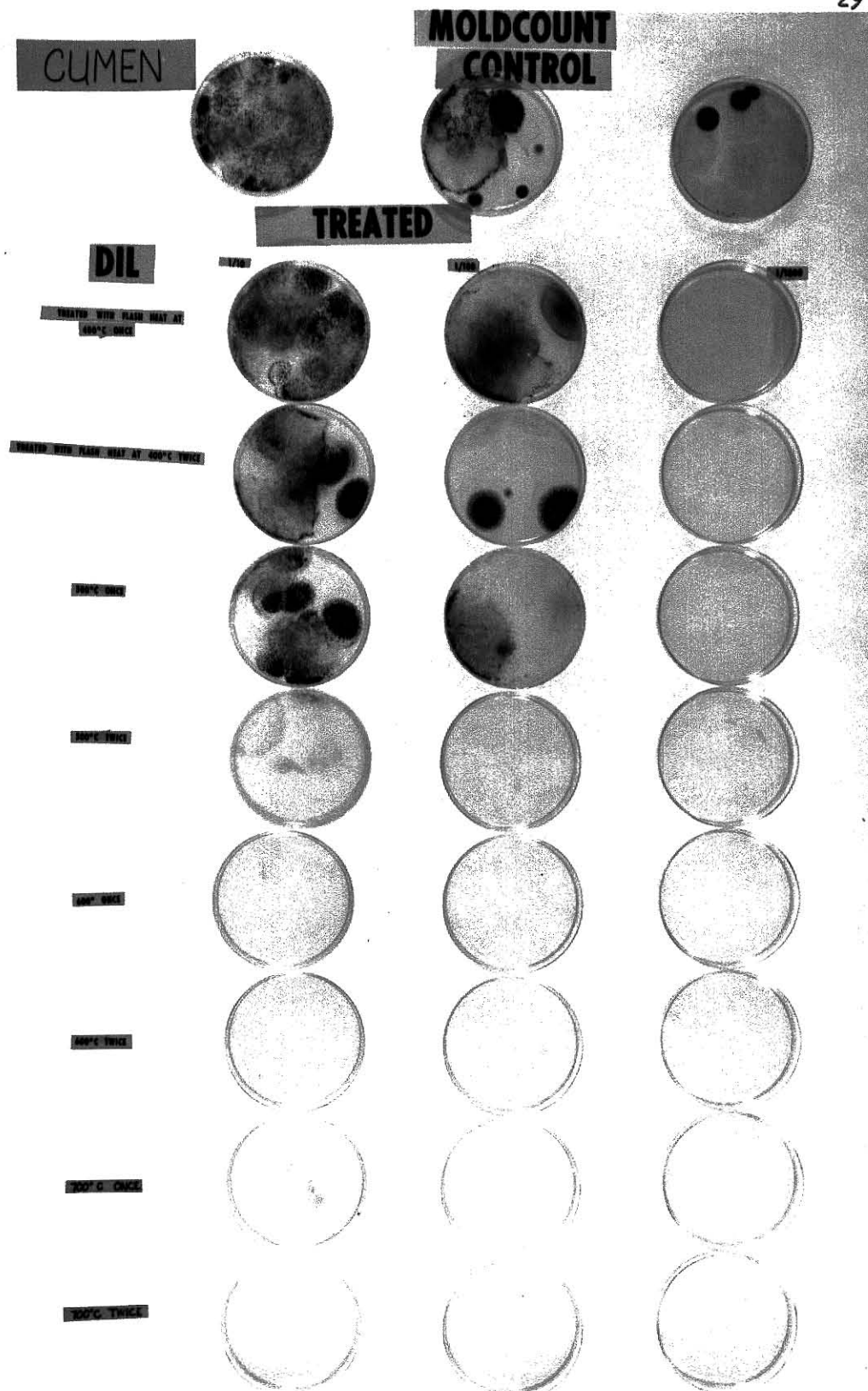


Figure IV. The number of mold before and after flash heat treatment.

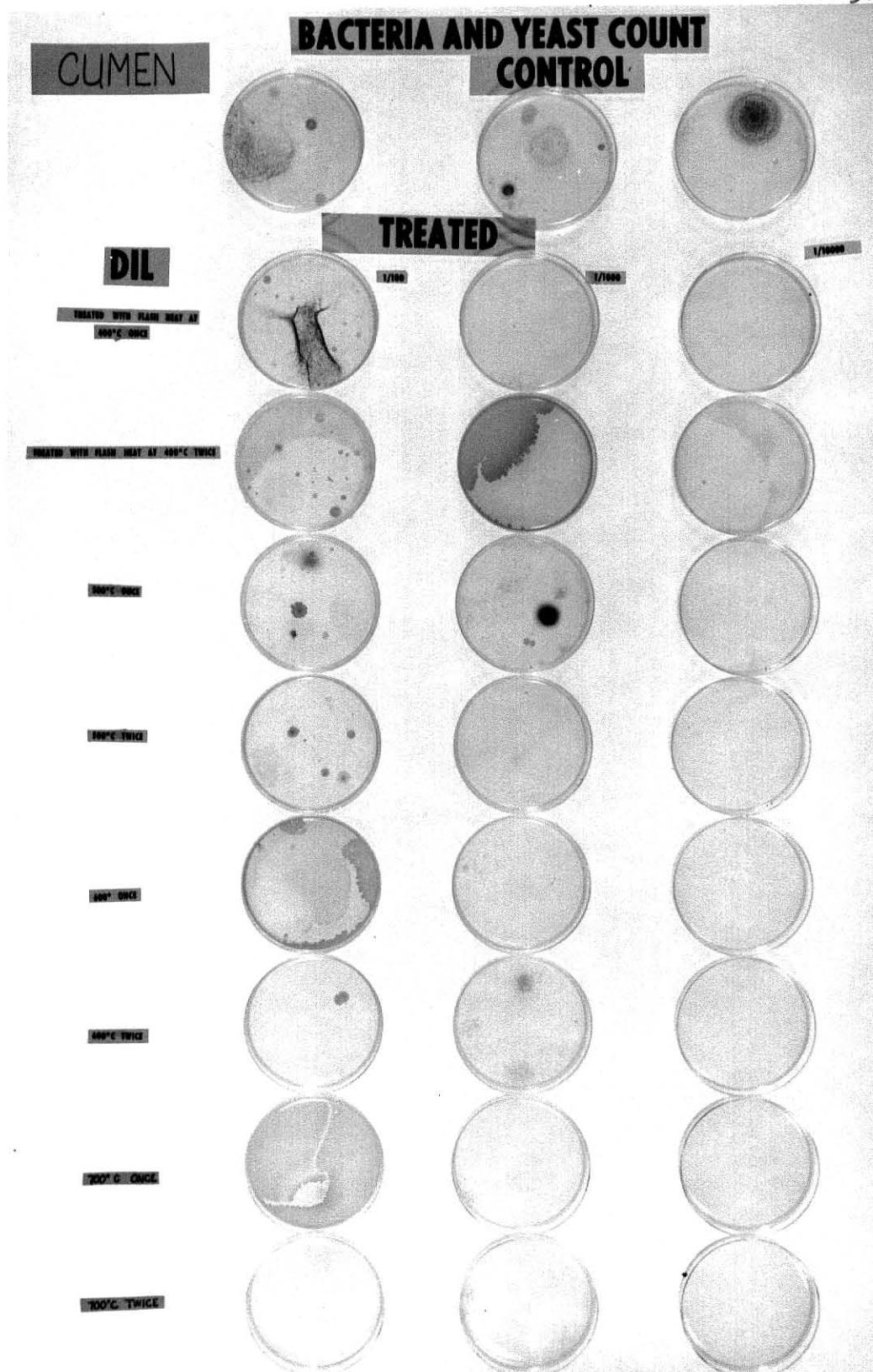


Figure V. The number of bacteria + yeast before and after treatment with flash heat.

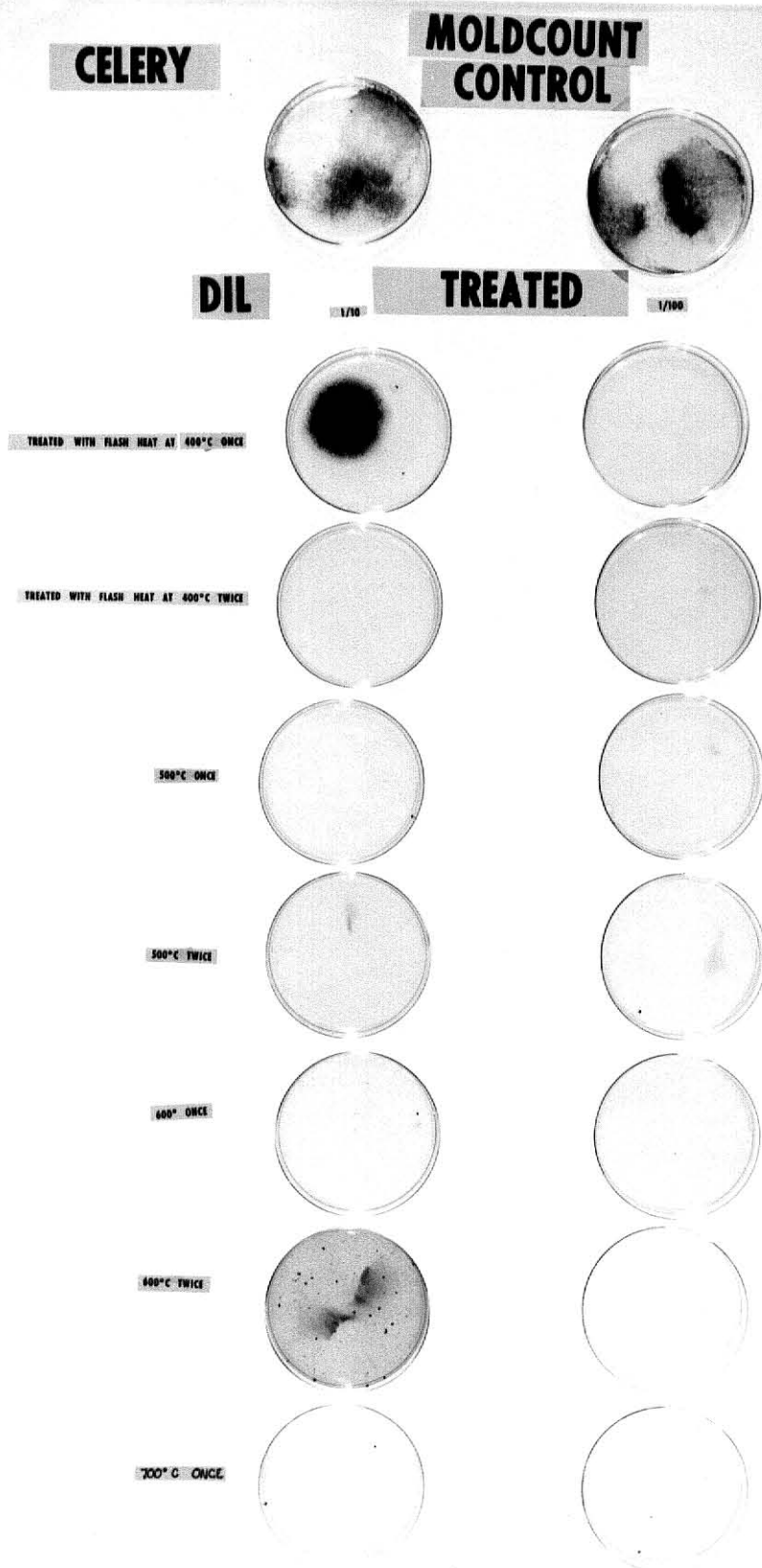


Figure VI. The number of mold before and after flash heat treatment.

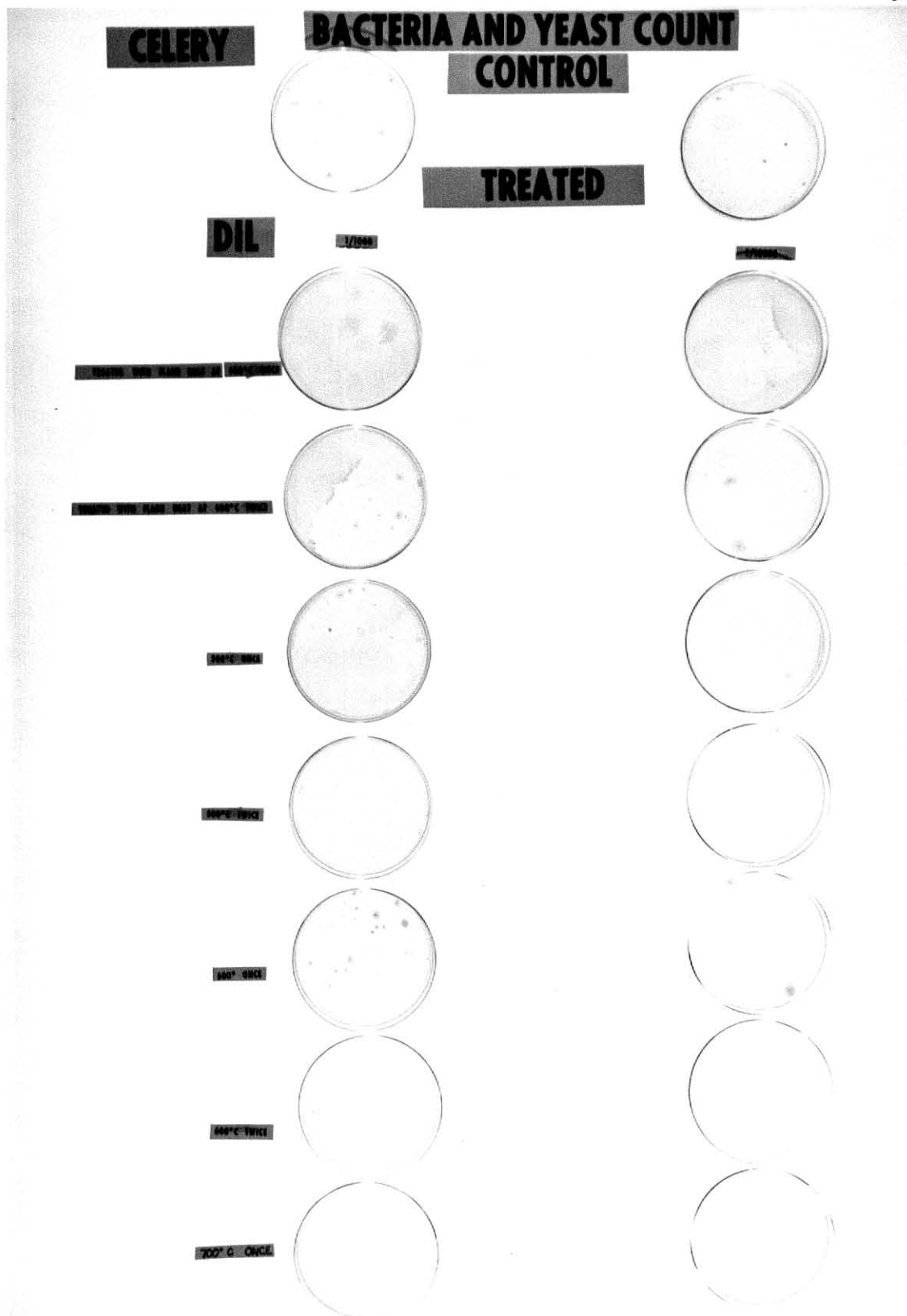


Figure VII. The number of bacteria + yeast before and after flash heat treatment.

air-cleaned sample of oregano. Since oregano is a leafy fine type of spice it could not be sanitized by flash heat treatment. Rather, it was necessary to treat with ethylene oxide to destroy the microorganisms.

The microbial count for the control sample was 300 colonies of mold per each gram of oregano while the bacteria + yeast count was 55 thousand colonies per gram. After oregano was subjected to ethylene oxide treatment for 5 minutes or more, the microbial population was reduced to zero. This is further illustrated by Figure IX.

Volatile Oil Content

The control sample of each spice and the samples representing the most drastic heat treatment in each case were analyzed for volatile oil content. In addition the recovered steam distilled volatile oils in each case were characterized for volatile constituents by gas chromatography.

Results of volatile oil determinations are as follows:

Sample	Volatile Oil
	%
<u>Fennel seed</u>	
A- control	2.00
Flash heat treatment at 700°C twice	1.31
<u>Black pepper</u>	
Control	3.36
Flash heat at 850°C twice	3.42
<u>Cumin</u>	
Control	2.50
Flash heat at 700°C twice	2.39
<u>Celery</u>	
Control	2.87
Flash heat at 600°C twice	1.78

These data suggest that some volatile oil is lost with the more drastic heat treatments. Black pepper and cumin are apparently little

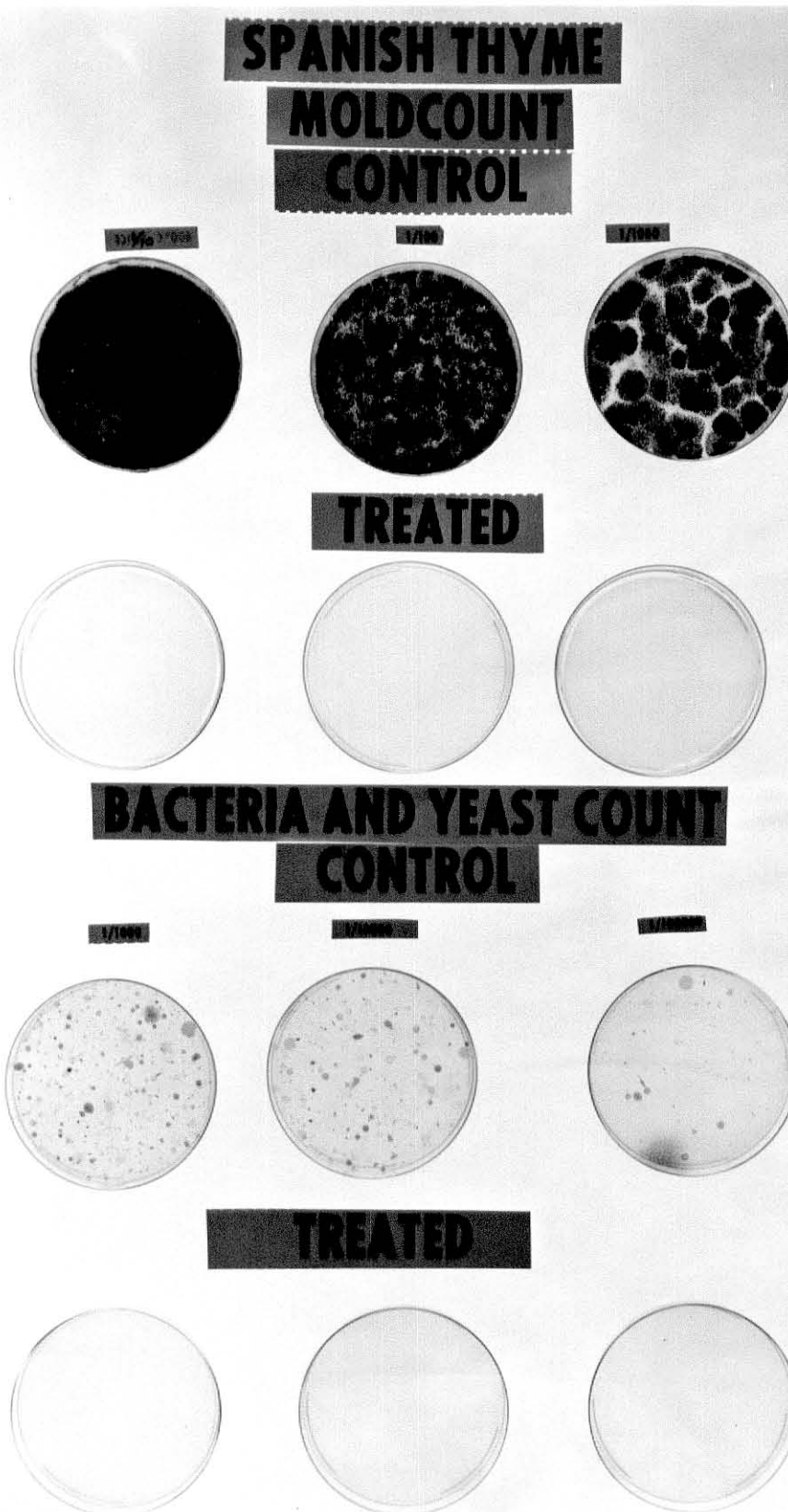


Figure VIII. The number of microorganisms before and after ethylene oxide treatment.

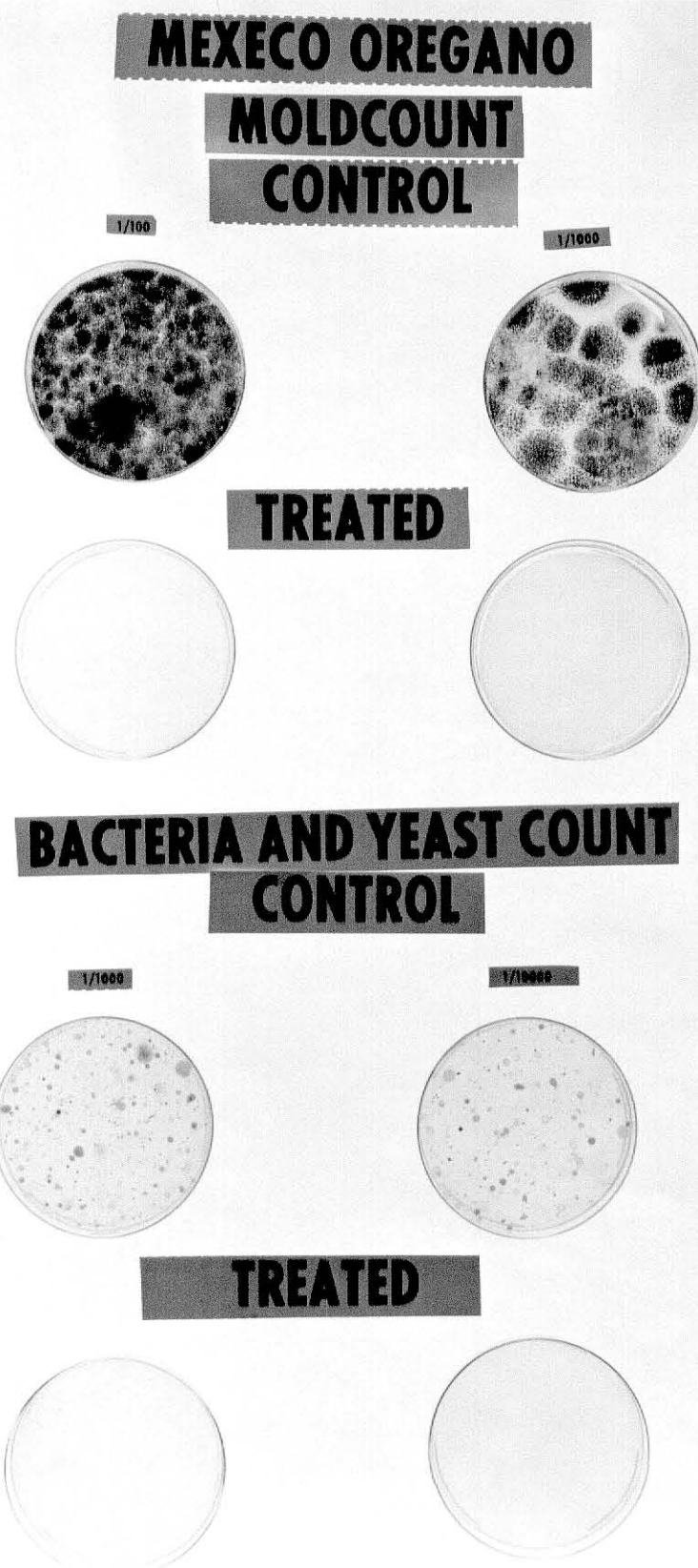


Figure IX. The number of microorganisms before and after ethylene oxide treatment.

affected by the heat treatment.

Gas chromatography profiles were performed on the controls and the following heat treated samples:

Fennel seed	Heat treatment	700°C	twice
Black pepper	"	850°C	"
Celery seed	"	600°C	once
Cumin	"	700°C	twice

In each, the G.C. profiles of the controls and the comparable heat treated samples were essentially identical.

Insect Contamination

The effect of heat treatment on spices infested with cigarette beetle are shown in Table 5.

Table 5. Cigarette Beetle Count

Treatment	No. per 2 Lbs.
Black pepper control	0
800°C once	0
Fennel control	4 larvae
600°C once	0
Cumin control	5 larvae
600°C once	0
Celery control	0
600°C once	0

These data illustrate that the strong odor of black pepper and celery destroyed the cigarette beetle even before it was treated with the flash heat. It was observed that flash heat incinerated the dead insects and debris. In the case of fennel and cumin, the cigarette beetle developed with storage but these were destroyed and removed upon flash heat treatment.

SUMMARY

Before sanitizing spice seeds, they were first cleaned by removing foreign materials by screening and aspiration. After mechanically cleaning the spices, they were treated with flash heat at various temperatures or by subjecting them with ethylene oxide.

Flash heat temperatures required to effectively remove molds were varied with the spice. For black pepper two treatments at 800°C was found most effective; for fennel two flash heat treatment at 600°C was effective; for cumin one exposure for 600°C was found effective while in celery seed two treatments at 400°C was effective.

Bacteria was destroyed at various flash heat treatments. Bacteria + yeast on black pepper were effectively destroyed by two treatments of flash heat at 850°C; in fennel one treatment at 700°C was effective; while in cumin and celery two treatments at 600°C was effective.

Treatment of thyme and oregano with ethylene oxide gas for five minutes was found effectively to destroy microorganisms.

ACKNOWLEDGMENTS

The author is sincerely grateful to Dr. John A. Johnson, major professor, for valuable advise and encouragement throughout this investigation and during preparation of this manuscript.

Gratitude is also extended to supervisory committee members Professor Arlin Ward and Dr. Paul Sieb and other members of the faculty and staff of the Grain Science and Industry Department, who willingly gave time, counsel, and advise during the course of this investigation.

Grateful acknowledgment is given to American Spice Traders Association for their financial support for this investigation.

Deep appreciation goes to my wife for her understanding, encouragement and help during this graduate work.

LITERATURE CITED

1. Rosengarten, Frederic. The Book of Spices. Wynnewood, Pennsylvania: Livingston Publishing Company, 1969.
2. Jank, Joseph K. Spices. St. Louis, U.S.A: Con.P. Curran Printing Co., 1915.
3. Jones, Lester W. (ed.). A Treasury of Spices. N.Y., New York: A.S.T.A., 1956.
4. Johnson, A. G. The Role of Fungi in the Deterioration of Grains. F.A.O. 88-91 Aug., 1947.
5. Milner, M. and Geddes, W. F. "Respiration and Heating" In Storage of Cereal Grains. A.A.C.C. 152-220, 1969.
6. Stuart, L. S. Federal Regulation of Bactericidal Chemicals Used in Industry. Jour, Milk & Food Tech. 25 (1): 308-312, 1962.
7. McCulloch, E. C. Disinfection and Sterilization. Philadelphia: Lea & Febiger, 1945.
8. Stewart, G. F. Introduction to Food Science and Technology. New York: Academic Press, 1973.
9. Bradley, C. L. Public Health Program. Jour, Milk & Food Tech. 25 (1): 138-140, 1962.
10. Richards, J. W. Introduction to Industrial Sterilization. London Academic Press, 1968.
11. Parker, M. E. Food Plant Sanitation. New York: McGraw Hill Book Co., 1948.
12. Baqir, A. W. Storage of High Moisture Wheat After Treatment With Flash Heat. K.S.U. Master Thesis, 1973.
13. Weiser, H. H. Practical Food Microbiology and Technology. A.V.I. Publishing Co., 1973.
14. Hags, H. Elliket. Effect of Acidification on Stability and Bactericidal Activity. Jour, Milk & Food Tech. 26 (5): 147-150, 1963.
15. Stevenson, K. R. Wet Grain Storage. Jour, Crops and Soils 24:8F, 1972.

16. Finch, W. H. Disinfectants, Their Values and Uses 180 P.P. London, Chapman & Hall, 1958.
17. Phillips, G. B. Industrial Sterilization. Duke University Press 4-10 PP., 1972.
18. Hale, W. S. and Sigmond Schwimmer and E. G. Bayfield. Studies on Treating Wheat with Ethylene. Cereal Chem. 20:224, 1943.
19. Balls, A. K. and Hale, W. S. The Effect of Ethylene. Cereal Chem. 17:490, 1940.
20. A Guide to Good Manufacturing Practices. Laughoff Grain Comp. Danville, Ill., 1973.
21. Parry, John W. Spices, Vol. I. New York, N. Y.: Chemical Publishing Company, 1969.
22. Parry, John W. Spices, Vol. II. New York, N. Y.: Chemical Publishing Company, 1969.
23. Knicker Bucker Mills. A Short Study of Spices. New York, N. Y.: Knickerbucker Mills, 1942.
24. Storbart, Tom. Herbs, Spices and Flavorings. New York, N. Y.: McGraw-Hill Book Company, 1973.
25. Hayes, E. S. Spices and Herbs Around the World. Garden City, N. Y.: Doubleday & Company, Ind., 1961.

Sanitizing Spice Seeds

by

Ayad Abdalla Ayad

B.SC., Alexandria University, 1965

An Abstract of a Master's Thesis

submitted in partial fulfillment of the

requirements for the degree

Master of Science

Department of Grain Science and Industry

KANSAS STATE UNIVERSITY

MANHATTAN, KANSAS

1975

Sanitizing spice seeds started by mechanical removal of foreign material from the spices by aspiration. Dust, sticks, chaff and other foreign matter were separated from spice seeds. Flash heat from a special apparatus developed at Kansas State University, Department of Grain Science and Industry, was used to remove microorganisms from black pepper, cumin, fennel and celery. Samples of spice seeds after aspiration were treated with flash heat at various temperatures, once and twice.

Microbial populations were determined by placing the sample in distilled sterile water, dilution, and plating on nutrient agar for bacteria and yeast and on potato dextrose agar for mold growth. Microbial counts showed remarkable decreases in molds, bacteria and yeast populations after treated with flash heat. Sanitizing as used here means removal of microorganisms normally found on the seed coat of spices.

Ethylene oxide was used to treat leafy spices, thyme and oregano. Ethylene oxide eliminated molds and bacteria.