FACTORS INFLUENCING THE FLAVORING OF PICKLES WITH SPICES AND ESSENTIAL OILS

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by

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Introduction

Spices are the chief source of flavors used in the pickle industry. With few exceptions the principal and characteristic flavor of a spice is contained in that component which is volatile with steam. This volatile component is termed the essential oil of the spice. When the spice is extracted in vinegar or combinations of vinegar and sugar, some of the essential oil is dissolved. The flavor is in this way made available to the pickles. The oils are practically insoluble in water, and only slightly soluble in acetic acid solutions. Therefore, it is almost a certainty that only a small portion of the spice oil is available when the spices are extracted in this way. Part I of this work is a determination of the extraction efficiency of vinegar solutions.

Essential oils from spices are often used, as such, to flavor pickles. The water insoluble oil may be incorporated into the flavoring solution by any one of the following three generally accepted methods: 1) A solution of oil in a solvent such as ethyl alcohol is added directly to the spicing liquor. 2) The oil is "cut" with sugar or salt. 3) An oil-water emulsion is prepared and added to the liquor. When organic solvents such as alcohols or sugar are used, the emulsion which forms upon the addition of the solution to vinegar is unstable. That is, the oil globules coalesce rapidly, rise to the surface of the spicing liquor and are partially lost by subsequent volatilization. Specially prepared emulsions are used to maintain the oil in suspension until the flavor is either dissolved by the pickle liquor, or absorbed by the pickle. Part II of this work concerns the

location of the spice oil in the spiced pickle. Some of the methods used to incorporate the oils in pickling solutions were studied in Part III.

Review of Literature

Analysis of Volatile Oil in Spices

Clevenger (1933) designed an apparatus for the determination of the volatile oil in spices by steam distillation. The apparatus consists of 500 to 2000 ml. short necked round bottom flasks, oil separatory traps and a Hopkins type condenser. Conditions found most suitable for distillation were also studied by Clevenger (1933, 1934, 1935). Lower yields indicate a lack of uniformity of mixing sample with the water, incomplete distillation or the loss of some volatile oil during the distillation. Conditions found most suitable for distillation of nutmeg and pimenta were as shown below.

Spice	Size of sample (gm)	Size of flask (ml.)	Volume of water (ml.)	Time (hrs.)
Nutmeg	25	500	200	4
Pimenta	100	1000	400	5

Results were compiled from the work of several collaborators. They were found to be quite consistent after the collaborators became familiar with the method. The method was adopted tentatively by the Association of Official Agricultural Chemists (1934) and officially for sage and marjoram analysis (1939). Cocking and Middleton (1935) developed a method similar to that of Clevenger for a determination of the volatile oil content of various drugs. The receiver which was used was designed for oils lighter than water. Turpentine was used as

a solvent for oils heavier than water. They found that in some cases better yields were obtained when the material was distilled in the whole form. Rowan and van Duuren (1937) compared several methods for the determination of volatile oil in spices and found that the method of Cocking and Middleton gave the most consistent results. Rowan and van Duuren modified the method slightly by using a 10 per cent sodium chloride solution instead of water in the distillation flask. They also used a special receiver for oil heavier than water. The samples were distilled until the amount of volatile oil remained constant.

Emulsions

Emulsions used in food industries are generally stabilized with gums such as acacia and tragacanth. In the pickle industry it has been convenient to add vinegar to the emulsion for the purpose of preserving it. The starches and gums would probably be attacked by bacteria and yeasts. This addition of acid to an emulsion may change the stability of the oil suspension as well as affect the gum stabilizers. Krantz and Gordon (1926, 1930) studied the effect of the hydrogen ion concentration on gum acacia and gum tragacanth emulsions. Emulsions containing 25 per cent cottonseed oil or Nujol (a mineral oil) were made. In 40 ml. of emulsion .5 gm. of gum tragacanth, or 2.5 gm. of gum acacia were used. The stable range of acacia was pH 2 - 10. Alkalies were detrimental. The globules were more uniform and smaller than in tragacanth emulsions. The stability range for tragacanth emulsions was pH 1.9 - 2.3. Rapid separation took place on the alkaline side. Clayton (1932) found that the viscosity of gum

tragacanth solutions was reduced by the addition of acid and sodium chloride. Limburg (1926) observed that the gelatin concentration required to stabilize a mineral oil-water emulsion was a function of pH value. His observations are shown in the table below.

Gelatin required (gm./liter)	pH
.00064	2.8
.00125	4.7
.01	5.5
.1	9.6

Hall (1934) in experiments with gum acacia and gum tragacanth emulsions concluded that the best pH range was 4.2 - 4.6 for emulsions containing one oz. of acacia and 1.5 oz. of tragacanth per gallon. Gray (1927) has shown that the addition of vinegar to mayonnaise emulsions does not cause the emulsion to break down, but merely dilutes the emulsion. Whitmore and Linehan (1929) added hydrochloric acid to five per cent orange oil gelatin emulsions and observed that the rate of oil separation was proportional to the acidity, once the emulsion had started to break.

It is known that particles of an emulsion carry an electric charge which aids in stablizing the emulsion by preventing coalescence. The addition of electrolytes affects the charge and often causes the emulsion to break. Clayton (1935) studied the effect of some inorganic salts on cylinder oil emulsions. Potassium chloride added to the emulsion increased the potential difference at first, but finally reduced the potential to nil at a concentration of 5000 millimoles per liter. Aluminum chloride, having a trivalent cation, reduced the potential to zero at a concentration of .51 millimoles per liter. Bhatnagar (1913) listed the coagulating efficiency of cations as Al > Cr > Ba > Sr > K > Na. It is particularly significant to suers of food

emulsions that the sodium ion has the least coagulating power. Gray (1927) observed that the addition of salt (NaCl) to an oil-water emulsion such as mayonnaise using egg yolk as an emulsifying agent causes the emulsion to be thicker and more permanent. He also found that small amounts of alum tended to break the emulsion.

Bennister and King (1938) found that sodium salts of sulphuric acid esters yielded oil-water emulsions which were relatively stable toward acids. This fact makes the use of sodium lauryl sulfonate desirable in the pickle industry, where acetic acid is used. However, experience has shown that it is well to be certain of the non-toxicity of any substance used in foods. This substance is widely used in dentifrices at concentrations varying from 1.25 to two per cent. Kitchin and Graham (1937) made observations on the effects of the sulfonates on the mouths of humans and dogs. They found no evidence of inflammation in either case. Epstein et al (1939) fed a sulfonate to rats and observed that, when daily intake was 50 grams per rat, the animals suffered an impairment of appetite. They conclude that the results were fairly conclusive as to lack of systemic toxicity. Correspondence with the Food and Drug Administration revealed that the use of sodium lauryl sulfonate must be made on the responsibility of the manufacturer. It is the understanding of the Administration that scientific work so far conducted has not shown that this chemical, when used in very small quantities in food products would be regarded as dangerous to health. As it would be used in pickle flavoring, the concentration would be less than .01 per cent.

Experimental

Part I - Extraction Efficiency of Vinegar Solutions

The solutions used for extracting the spices were 130 grain vinegar (13% acetic acid) and sucrose solution in 130 grain vinegar. The spices used were whole cloves, whole dill seed, cassia quills and nutmegs. Cloves and dill seed were used in the whole form in the extraction and subsequent distillation. The cassia quills and whole nutmegs were broken in a food grinder immediately before the extraction. Dill seed was extracted with 130 grain vinegar only.

The apparatus used to determine the volatile oil in the spices is shown in Figure 1. It is the same as the one recommended by the Association of Official Agricultural Chemists (1934, 1939) for spices. Preliminary distillations were run on unextracted spices in which the effects of time, temperature of bath, quantity of water used, the size of flask and the condition of the spice were studied. The effect of the length of time of distillation was different for different spices as is shown in Table 1.

It was found that the maximum temperature to which the oil bath could be raised without loss of some volatile oil was 125°C. - 130°C. (257°F. - 266°F.). This temperature was optimum for the smaller flasks, 250, 500 and 1000 ml., when they were submerged to slightly above the water level in the flask. However, it was necessary to submerge a 2000 ml. flask to slightly below water level at these temperatures. In order to prevent loss of some of the volatile oil, it was found necessary to rest the tip of the condenser (Figure 2a)

Table 1 Per Cent Volatile Oil Obtained from Spices After Distilling for Various Lengths of Time.

Spice	Per cent oil		
	5 hours	8 hours	11 hours
Allspice	2.7	3.0	3.0
Cassia	1.0	1.1	1.1
Cinnamon	1.0	1.1	1.1
Cloves	14.2	17.0	18.4

Table 2 Per cent Volatile Oil Obtained From Spices by Different Investigations

SPICE	PRESENT STUDY	Reported by	
		GLEVENGER	GILDEMEISTER
Allspice	2.7 - 3.0	2.7 - 4.3	3.0 - 4.5
Cloves	17.0 - 18.4	18.0 - 18.2	16.0 - 19.0
Cassia	1.15	1.5 - 4.0	1.2
Dill Seed	2.9		2.0 - 4.0
Nutmegs	9.4	6.8 - 8.7	7.0 - 15.0
Cinnamon	1.1	3.2 - 3.3	0.5 - 1.0

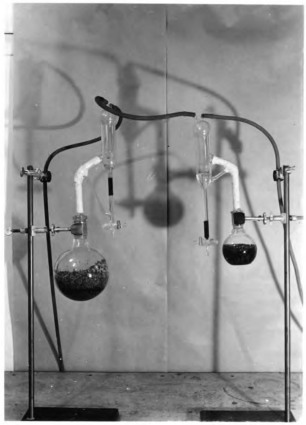

Figure 1

The Clevenger apparatus showing traps for oils lighter than water (left) and for oils heavier than water (right).

Figure 2

The tip of the condenser (a) is resting against the opening of the distillation tube at (b).

against the opening of the distillation tube (Figure 2b). Although most of the volatile oils are slightly soluble in water, the volume of water used could be varied considerably without changing the amount of volatile oil produced. For example, 25 grams of cloves distilled with 65 ml. of water produced exactly 4.6 ml. of oil, as did 25 grams distilled with 135 ml. of water. However, 15 hours of distillation were required to obtain the oil from the flask containing 65 ml. of water, while only 10 hours were necessary to distill all of the oil from the flask containing 135 ml. of water.

The size of the flask used was determined by the amount of oil present in the spice since it was necessary to use a larger amount of some spices, such as cassia or dill to obtain a sufficient quantity of oil to measure. To determine the effect of the size of the flask on the yield of volatile oils, whole cloves were distilled in two different sizes of flasks - 250 ml. and 500 ml. The amount of oil distilled was 18.4 per cent from each flask in 12 and 14 hours.

When finely ground spice was used, there was a tendency for it to stick to the bottom of the flasks and become charred. Whole or broken spice did not do this.

This Clevenger method was used throughout the experiemtn to determine the volatile oil, first in unextracted spices, and then in extracted spices. The per cent volatile oil in various unextracted spices as determined in this experiment is shown in Table 2, along with those reported by Clevenger et al (1933, 1934, 1939, 1941) and Gildemeister and Hoffman (1916).

Extraction Efficiency of Vinegar

A study was also made of the extraction efficiency of vinegar under certain conditions. The influence of time and temperature and the addition of sugar to vinegar upon the amount of volatile oil extracted from spices was determined. Extractions were made on whole cloves with 130 grain vinegar and 170 grain vinegar - 25 per cent sugar solutions at 200°F. and at 150°F. The extraction at 200°F. was made by heating the flask containing the spice and solution to 200°F. and allowing it to cool immediately. The extraction mixtures at 150°F. were held at that temperature for four hours in a constant temperature water bath. Samples were allowed to stand one, seven and 21 days before distillation. Broken cassia and nutmegs were extracted in similar solutions at 150°F. only, for four hours. The nutmegs stood in the extracting solutions one, 14 and 60 days before distillation. The dill seed was extracted in 130 grain vinegar and 65 grain vinegar at 200°F. for three hours. (Tables 3 and 4)

At the proper time the extracting solutions were poured off, the spices were washed with two portions of cold water and then were distilled. The oil which was obtained in this distillation had not been extracted from the spice by the vinegar solution. In Table 3 are shown the amounts of oil in per cent extracted by the solutions under all conditions. The effect of time of standing upon the extraction efficiency was not consistent. Under one set of conditions, for example, the per cent oil extracted from cloves increased from 15 to 20 per cent in seven days, but actually decreased to eight per cent in 21 days. When the effect of temperature and the addition of sugar were

Table 3 Essential Oil Extracted From Spices by Vinegar Solutions

Spice	Number of days extracted	Temperature Treatment	Per cent volatile oil			
			Original spice	Left in spice after extraction	Removed by vinegar each extraction	Removed by vinegar and sugar at each extraction
Cloves	1	200°F.*	17.0	14.4	2.6	
"	7	"		13.6	3.4	
"	21	"		15.6	1.4	
Cloves	1	150°F.**	17.0	12.0	5.0	
"	7	"		16.0	1.0	
"	21	"		15.2	1.8	
Cloves	1	200°F.*	17.0	12.8		4.2
"	7	"		14.4		2.6
"	21	"		16.8		.2
Cloves	1	150°F.**	17.0	15.6		1.4
"	7	"		14.4		2.6
"	21	"		16.0		1.0
Cassia	1	150°F.**	1.2	.3	.9	
"	7	"		.4	.8	
"	21	"		.3	.9	
Cassia	1	"	1.2	.5		.7
"	7	"		.4		.8
"	21	"		.3		.9
Nutmegs	1	150°F.**	9.4	6.8	2.6	
"	14	"		6.8	2.6	
"	60	"		7.6	1.8	
Nutmegs	14	"	9.4	6.4		3.0
"	60	"		7.6		1.8
Dill seed	1	200°F.*	2.9	2.8	.1	
" "	1	"		2.9	0	

* Temperature of mixture brought to 200°F. after which spices stood in extracting solution at room temperature for number of days indicated in Column 2.

** Mixture heated at 150°F. for four hours after which spices stood in extracting solution for number of days indicated in Column 2.

Table 4 The Influence of Temperature and Addition of Sucrose on the Extraction Efficiency of Vinegar

Spice	Solution	Temperature	Per cent of total oil extracted
Cloves	130 gr. vinegar	200°F.	14.7
"	"	150°F.	15.0
"	25% sucrose in 130 gr. vinegar	200°F.	13.5
"	"	150°F.	10.0
Cinnamon	130 gr. vinegar	150°F.	73
"	130 gr. vinegar with sucrose	150°F.	67
Nutmegs	130 gr. vinegar	150°F.	25
"	130 gr. vinegar with sucrose	150°F.	26

studied, it was observed that, while temperature alone did not appreciably affect the amount of oil extracted, the addition of sugar decreased the amount of oil extracted from cloves and cinnamon, but slightly increased the amount extracted from nutmegs as shown in Table 4.

An experiment was done to determine the influence of repeated extraction of cloves with 130 grain vinegar. Seven flasks containing 25 grams of whole cloves and 100 ml. of 130 grain vinegar were heated in a steamer to 200°F. after which they were allowed to stand before the vinegar was changed, or the spice was distilled. Each time new vinegar was put on the spice, 70 ml. were used, since the dry spice had absorbed and retained 30 ml. from the previous addition. The results are shown in Table 5.

In addition to the work above, three spices, dill herb, allspice, and cassia, were picked out of a mixture of whole spices consisting of mustard and celery seed, bay leaves, chili peppers, cardamom, ginger root, allspice and cassia (with the latter two predominating), which had been used to make genuine dill pickles at a pickle plant. These three spices were tested for their volatile oil content. The results are shown below.

Spice	Per cent volatile oil	
	After use in making dill pickles	Normally present in unused spice
Dill herb	.8	0.25 - 0.4 *
Allspice	1.6	3.0
Cassia	1.3	1.2

* Yield from commercial steam distillation

However, the odor of the oils obtained from these spices indicated that all the distillate obtained from the dill herb was not dill oil, but rather a mixture of spice oils, indicative that the dill herb

Table 5 Per cent of Volatile Oil Extracted from Cloves by Repeated Extractions with 17% Ethyl Alcohol

Number of times extracted	Total number of days extracted	Per cent volatile oil left in cloves	Per cent removed by this extraction	Total per cent extracted
0		18.4	0	0
1	1	16.8	1.6	6.5
2	2	16.8	0	6.5
3	3	16.8	0	6.5
4	4	15.2	1.6	17.5
5	10	14.4	0.8	22.2
7	15	12.8	1.6	28.2
9	21	9.6	3.2	47.8

acted as an excellent absorbent for spice oils.

Part II - The Location and Mechanism of Absorption of Volatile Oil by Pickles

Whether the essential oils are extracted from the spices or added, as such, to the flavoring solution, they occur as small globules in the pickling brine. A preliminary experiment was performed in order to determine the form and location of these oil globules in spiced pickles. To 50 gallons of pickles which had been sweetened, but not spiced, was added enough spice oil, in emulsion form, to make the final concentration 1 part oil to 5000 parts of pickle and liquor. After several weeks, samples of the liquor were examined microscopically for the presence of oil globules. None were found. A 1:5000 dilution of spice oil in a fresh vinegar-sugar solution of the same composition, contained oil globules which were easily found. Assuming a drop of the liquor containing 1:5000 parts of oil to have a volume of 0.05 ml., and the size of the oil globules to have a diameter of 50 microns, then each drop of liquor would contain 153 globules of oil. If the oil globules were 10 microns in diameter, there would be 20,000 per drop. Since globules of this size would be easily discernable under the microscope, it was assumed that they had been either absorbed by the pickles or had evaporated.

It is well known that volatile oils are readily absorbed by some ether soluble substances. A determination of crude fat was made on the epidermis, parenchyma and the central portion (placental tissue and the seeds) of processed-unspiced pickles. The epidermis was scraped from the pickle with a sharp knife, and the placental tissue

and seeds were cut and scraped from the remaining parenchyma. These tissues were then dried in flowing hot air at 195°F. for 12 hours, cooled in a desiccator and weighed into a Soxhlet extraction thimble. The Soxhlet apparatus used for the determination of ether extract is shown in Figure 3. Anhydrous ethyl ether was used as the solvent. Two sizes of pickles were used for the extraction, namely, 4500-6000's and 1000's. * It was found that in the smaller pickles the central portion contained the greatest amount of crude fat, while in the larger pickles, the epidermis contained most of the fat. This is shown in Table 6.

The location of ether soluble constituents having been determined, stains were made on whole pickles, cut pickles and lamellar sections, and viewed under microscopes having magnifications from 9x to 450x. It was thought that, if the spice oil which had entered the pickle were given some color, it would be seen more easily under the microscope. The stain used for this purpose was Sudan IV, which is a red, oil soluble dye. The following three procedures were used.

1) A pickle was allowed to stand in a concentrated solution of Sudan IV in dill oil for one week. When lamellar sections of this pickle were observed under the microscope, it was observed that the epidermal layer of palisade cells was stained a very deep red. The skin at the blossom end of the pickle was not as deeply stained as that at the stem end. No internal areas were stained.

2) A cut pickle was placed in a Sudan IV - dill oil mixture for 24 hours. The oil penetrated fairly well into the exposed central por-

* Pickle size is expressed as the number of pickles contained in a 45 gallon barrel.

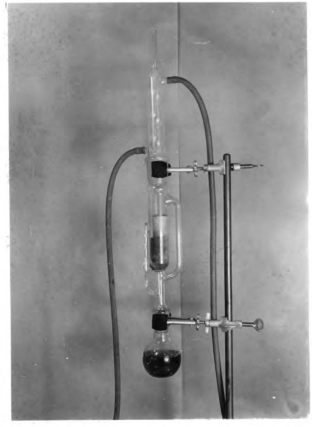

Figure 3

The Soxhlet apparatus used for the determination of ether extract.

Table 6 Ether Extract of Epidermis, Parenchyma and Central Portion of Processed Pickles.

Size of pickles	Section	Per cent of whole pickle	Per cent ether extract	Per cent of total ether extract of pickle
4500-6000	Epidermis	30	3.5	38.5
	Parenchyma	53	1.9	36.5
	Central portion	17	4.0	25.0
1000	Epidermis	11	4.4	31.3
	Parenchyma	59	1.2	45.5
	Central portion	30	1.2	23.0

tion, and was concentrated in the epidermis, but was not apparent in the interior of the parenchymatous cells.

3) Dried lamellar sections were put into this Sudan IV - dill oil solution for 24 hours, washed in 95 per cent alcohol for 30 seconds, blotted and dried. Similar sections were stained in alcoholic Sudan IV. The skin and vascular ducts in the center portion of the control pickle were stained lightly. The pickle which had been in the dill oil was stained heavily in the vascular ducts, in the nuclei of the cells just beneath the epidermis, as well as in the epidermis itself.

The above facts led to an experiment to determine the volatile oil content of pickles and pickle sections which had been treated with relatively concentrated dill oil emulsions. Dill oil was used for two reasons, it is very insoluble in water, and it is readily available.

For this purpose salt stock pickles were processed, all of the salt being removed. To these pickles enough water and dill oil emulsion were added to give the desired final concentration of oil. Two different sizes of pickles were used, 6000 - 7500's and 1200 - 1600's. Final concentrations of oil were 1 part to 100 parts pickle and brine, 1:200, 1:1000 and 1:2000. The ratio of pickles to brine was 5:3. After the pickles had remained in these solutions for three days, they were removed, washed, ground and distilled in the Clevenger apparatus for the determination of volatile oil content. A similar distillation of unspiced pickles revealed that they contained less than .05 per cent volatile oil. The results as shown in Table 7 indicate that the size of the pickle did not influence the amount of oil absorbed, and that a greater per cent of oil was absorbed in the higher dilutions. Under

Table 7 The Relation Between the Concentration of Dill Oil present and the Amount Absorbed by Processed Pickles.

Ratio of oil to pickles and liquor	Size of pickles	Total ml. of oil added	Ml. oil absorbed	Per cent of oil absorbed - based on oil absorbed from 1:100 dil.	Per cent absorbed of amount present in brine
1 : 100	6000 - 7500	8.0	1.10	100	14
1 : 200	" "	4.0	1.00	90	25
1 : 1000	" "	.8	.50	45	63
1 : 100	1200 - 1600	8.0	1.45	100	18
1 : 200	" "	4.0	1.20	83	30
1 : 1000	" "	.8	.50	34	63
1 : 2000	" "	.4	.22 - .25	17	56 - 63

the conditions of this experiment, the pickles absorbed a maximum of 63 per cent of the oil added to the spicing liquor.

In the determination of the location of the volatile oil in flavored pickles (1000 size) two different procedures were used. In one set the pickles were peeled before they were spiced, and in the other set the pickles were peeled after they were spiced. The concentration of spice oil used was 1:200. The ratio of pickles to brine was 2:1. The results of this experiment are shown in Table 8. The data show that most of the oil is absorbed by the epidermis of the pickle.

This work was continued to determine how much of the oil in the pickle was actually in solution. Pickles were treated as before with emulsions of dill oil. The ratio of pickle to brine in this experiment was 5:3. As much fluid as possible was removed from the pickles in a hand press, and the pulp from the pickles distilled in the Clevenger apparatus to determine the amount of oil retained in the pulp. The results shown in Table 3 indicate that most of the oil was retained within the solid portion of the pickle. The data in Tables 6, 7 and 8 would indicate not only that the greater amount of dill oil was absorbed by the pickle from the liquid, but that it was dissolved in those tissues of the pickle containing the greatest amount of lipids. Since 37 per cent of the pickle was removed as juice, 63 per cent of the pickle actually held 89 per cent of the oil absorbed by the whole pickle.

Table 8 The Location of the Dill Oil Absorbed by 1000 Size Pickles

Section	Per cent of whole pickle	Per cent of total oil absorbed
Epidermis	27	53 - 58
Parenchyma	17	27 - 29
Central portion	56	15 - 18
Pressed pulp	63	89

Part III A Study of Methods of Incorporation of Spice Oil in Pickles.

Although this work is incomplete, the experiments here described may assist in a further study of the problems involved in pickle spicing. Four methods of incorporating oil of dill in spicing liquors were studied, namely the addition of - 1) an alcohol solution of oil, 2) an emulsion, 3) an oil-sugar mixture, and 4) the plain oil itself. In each case 3.6 ml. of oil of dill was added to a gallon jug containing 2200 ml. of processed pickles and 1400 ml. of an acidified water. The oil in alcohol solution used was 1 part oil in 9 parts of alcohol. A gum arabic emulsion was used containing 25 per cent oil and 10 per cent gum. Duponol PC, a surface tension depressant in which the active ingredient is sodium lauryl sulfonate, was added to one of the samples in which the palin oil was incorporated. The sugar-oil mixture was made by the addition of 3.6 ml. of oil to 400 gms. of sugar, and the addition of this mixture to 550 ml. of 136 grain vinegar and water to make 1400 ml. . After the sugar was dissolved, the liquor was added to the jug of pickles. Trichloracetic acid was used to acidify the samples not containing vinegar. The jugs were inverted and shaken daily. After two weeks the pickles were drained, ground with 1000 ml. of water and distilled in the Clevenger apparatus. The results shown in Table 9 indicate that under the conditions of this experiment, no one method incorporated very much more oil than any other method. It was observed during the course of the experiemnt, however, that the oil came to the surface of the brine in all of the jugs except the one in which the emulsion was used.

An attempt was made to determine how much of the oil added in

Table 9 Per Cent of Oil of Dill Absorbed by Pickles From Acidified Solutions (1 part oil - 1000 pickles and brine)

Method of incorporation	Per cent absorbed
Alcohol solution	61
Emulsion	55
Plain oil	62
Plain oil with Duponol PC	55
Sugar-vinegar	63

alcoholic solution to various types of pickling liquors would return to the surface of the liquor. A 10 per cent dill oil in alcohol solution was added to - 1) water, 2) 136 grain vinegar, 3) 50 per cent sucrose in 136 grain vinegar, 4) 20 per cent sodium chloride in 136 grain vinegar, and 5) 50 per cent sucrose and 15 per cent sodium chloride in 136 grain vinegar. Ten milliliters of the alcoholic solution were added to 90 ml. of the above solutions in a 110 ml. cassia flask. The mixtures were shaken and allowed to stand until oil was visible on the surface of the solution. The oil layer was brought up into the neck of the flask by the addition of the respective solutions, where it was measured. The results are shown in Table 10. The salt-sucrose-vinegar solution seemed to retain the oil better for a period of two days, after which there was a noticable increase in the amount of oil separated.

Table 10 The Stability of One Per Cent Alcohol - Dill Oil Emulsions in Various Types of Pickling Brines

Type of brine	Per cent oil emulsified	At end of
Water	10	5 minutes
136 grain vinegar	20	15 minutes
50% sucrose in 136 grain vinegar	30	15 minutes
20% NaCl in 136 grain vinegar	15	5 minutes
50% sucrose and 15% salt in 136 Grain vinegar	45	30 minutes
"	40	16 hours
"	15	48 hours

Summary

1. The greatest amount of oil was removed from the spices in 24 hours after which time the oil was dissolved from the spices at a much slower rate under the conditions of the treatment used.

2. The addition of sugar to vinegar decreased slightly the amount of oil extracted from cloves and cinnamon, but had little effect on nutmeg.

3. Vinegar is a poor solvent for clove oil, since nine extractions, over a period of 21 days, removed only 47.8 per cent of the oil from the cloves. This indicates a tremendous loss of oil by the use of whole cloves in pickle manufacture.

4. Processed pickles absorbed from 56 to 63 per cent dill oil from a spicing brine containing a 1:1000 and 1:2000 dilution of the oil.

5. A greater amount of oil was absorbed from processed pickles from the higher than from the lower concentrations. However, a relatively greater per cent of oil was absorbed from the lower than from the higher concentrations. In the stronger concentrations (1:100 and 1:200), the amount of oil absorbed was not directly proportional to the amount of oil present, while at the weaker concentrations (1:1000 and 1:2000) it was directly proportional.

6. The per cent of ether extract varied inversely with the size of the pickle, the smaller pickles having a greater amount than the larger. There was a variation of the amount of ether extract in the same type of tissue in different size pickles.

7. The different methods tried so far of incorporating a 1:1000 dilution of dill oil into pickles within a period of two weeks showed

little difference in the amount absorbed.

8. An emulsion resulting from the addition of a 10 per cent solution of dill oil in 95 per cent alcohol to various types of pickling solutions showed the greatest stability in a brine containing 50 per cent sucrose, 15 per cent salt and 136 grain vinegar, and the least stability in a brine containing 20 per cent salt and 136 grain vinegar.

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