AN ABSTRACT OF THE THESIS OF

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Title: ESSENCE RECOVERY FROM STRAWBERRY POMACE

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The intensity and quality of essences distilled from strawberry pomace, obtained from a juice concentrating plant, were studied. Analysis of variance of sensory panel scores indicated that incubation temperature of the pomace prior to distillation affected the intensity and quality of essence aroma more than holding time or its pH. Intensity showed a marked decrease as the pH of the pomace was increased above 4.0 prior to distillation. The panel was able to detect statistically significant differences ($p<.01$) in the quality of aroma of essences obtained from pomace processed above pH 4.0 and held at 20 to 50°C. Optimum essence production was detected in pomace adjusted to pH 4.0, prior to distillation and held at 40°C for 4 hr or longer. Gas chromatographic (GC) analysis of several essences collected from strawberry pomace processed under different conditions revealed dissimilar patterns. Essences of highest quality, as determined by the sensory panel, had GC patterns similar to that of essence produced from whole strawberries. All pomace essences contained high

concentrations of benzaldehyde, 2-heptenal, ethyl hexanoate, limonene, 2-octenal, linalool, benzyl acteate, and ethyl cinnamate. Presence of these compounds may account for the reduced quality of essence produced from strawberry pomace.

Essence Recovery from Strawberry Pomace

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REVIEW OF LITERATURE

Utilization of Food Processing Waste

With ever increasing energy cost and growing populations, food scientist have searched for more efficient processing methods and alternative food sources. Problems associated with waste generated by food industries such as, disposal and energy cost, environmental impact and discarding nutrients is a special concern in the United States. Producing a useable product from this waste would help stem increasing cost and produce food for growing populations. For example, brewery and distillery waste, which contain a large proportion of vitamins and proteins are currently dried and sold to commercial feed mixers (Lipsett, 1963). In light of the high energy cost involved in drying processes, an alternative process would involve extraction of the nutrients from the spent mash residue to be used in fortifying purposes.

Perhaps the main consideration when evaluating whether to utilize food processing waste are oriented towards economical and marketing prospects rather than needs for new technology. However, some very successful applications of food waste utilization are currently used in the United States. By-products from the pickling and processing of

fruits were used to produce vinegars, flavors, yeast, vitamin products, drugs, oils, syrups, paints and paper as well as feed for livestock (Lipsett, 1963).

New Approaches to Food Waste Utilization

As shortages keep reoccuring and raw materials become more scarce, experimentation into the utilization of food waste have become more creative. Berrh (1976) reported on a method of recovering mango juice from the peel and waste fibrous pulp by treatment with pectic enzymes. The utilization of tomato cannery waste, to recover tomato protein concentrate, was described by Kramer and Kwee (1977). Citrus fruit industries have been plagued by oils and suspended solid material in their waste water causing a high biochemical oxygen demand (BOD). Essential oils and suspended solid matter have been recovered from lemon processing waste water by combined solvent extraction and emulsion flotation. The result was a potentially valuable product and reduction of the BOD (Earkart and King 1976). A novel approach for the growth of various molluscs filter feeders in an aquaculture system was documented by Mann and Ryther (1977). Experimental animals were fed algae grown on secondary treatment sewage effluent. Two of six species of bivalve molluscs exhibited an increased live and dry weight.

The utilization of unedible plant material by fermentation processes is a current area of interest. Production of single cell proteins and lipids by action of various microorganisms on food processing waste was the topic of a recent symposium (Birch et al., 1976). Forster et al. (1977) reported the potential for fermentation of food processing waste of plant material and summarizes the raw materials needed, pretreatment methods, microorganisms and value of the end-products.

The environmental impact of whey disposal has made the dairy industry well aware of the significance of utilizing whey as a food or feed product to minimize pollution. In 1974 approximately 20% of all milk processed in the United States was used in cheese making, which resulted in whey of one type or another (Jones 1974). Jones stated

> one of the most promising developments in respect to whey utilization lies in the recovery of the protein fraction, which has unique nutritional and functional properties. The nutritional value of whey protein exceeds that of casein; the protein has unique whipping and emulsifying properties. Removal of the protein from whey reduces the whey solids about 13% and the BOD about 20%.

Utilization of Waste for Essence Recovery

Increasing the strength of natural essences by the utilization or manipulation of fruit processing by-products has not been studied extensively. Because of the high

market value for fruit essences, research into this area may offer valuable returns. Guadagni et al. (1971a) reported that the components associated with apple aroma, most notably esters, increased significantly when held at 22°C for 1-2 days. Moreover, apple volatile production was several times greater in the peel than in fresh or whole apples. Guadagni et al. (1971b) evaluated the intensity and quality of essence derived from apple peels against standard apple essences from whole apples and unconditioned apple peels. The aroma recovered from peels held at ambient temperature (19-23°C) for 24 hour was 2-7 times stronger than from unconditioned peels. Guadagni (1971b) stated that,

> Quality of the aroma solution from conditioned peels was statistically indistinguishable from standard aroma solutions. Thus, the conditioned peels gave aroma solutions with 2-7 times more 'flavoring capacity' than no-delay peels, without significant sacrifice in quality.

Investigations in the recovery of fruit essence from pomace has not been reported in the United States. However, an interesting paper by Karwowska et al. (1969a) described the production of essence from blackberry pomace. Using pilot plant equipment to produce essences from the pulp, juice and pomace of blackberries, these authors found the essence obtained from the pomace was "most intensive and characteristic of blackberry aroma." Although none of

the compounds contributing to the aroma of blackberries were identified, gas chromatographic (GC) analysis revealed 36 peaks from the presscake, 22 peaks from the pulp and 20 peaks from the juice. Karwowska et al. (1969a) later looked at various combinations of the three extracts and found "the richest aroma was obtained by combining the extracts from fruit pulp pomace and juice." Specifications were developed on an industrial scale for producing essence from blackberry pomace.

Composition of Strawberry Essence

The aroma constituents of strawberries have been thoroughly investigated. Early investigations, using various chemical and chromatographic means for identifications, indicated that strawberry aroma is a complex mixture (Coppens and Hoejenbos 1939, Winter 1958, Seidel et al. 1958, Willhalm et al. 1966).

With advent of (GC) in conjunction with mass spectrometry (MS), infrared spectrometry and nuclear magnetic resonance, over 150 compounds have been separated and identified (Teranishi et al. 1963, McFadden et al. 1965, Tressl et al. 1969). These have been summarized by Nursten (1970) according to the class of the compound. In all 12 acids, 27 alcohols, 52 esters, 21 carbonyls, 20 acetals and 10 hydrocarbons were classified. Recently, Mussinan and Walradt (1975) extracted 33 volatile acids from fresh strawberries, of which 18 had not been previously reported in strawberries.

A list of volatiles reported in strawberries is contained in the following table.

Table 1A - Compounds previously identified in strawberries.

Acids (b)

Propionic
Butyric
Isobutyric
2-methylbutyric
Valeric
Isovaleric
Hexanoic
Isohexanoic
Heptanoic
Isoheptanoic
Octanoic
Nonanoic

Decanoic
Undecanoic
Dodecanoic
Palmitic
2-Methyl-2-butenoic
Methylbutenedioic
2-Methyl-cis-3-pentenoic
2-Methyl-trans-3-pentenoic
2-Methyl-2-pentenoic
2-Hexenoic
2-Octenoic
3-Nonenoic

3-Hydroxyhexanoic
3-Hydroxyoctanoic
Phenylacetic
Phenylpropionic
Benzoic
4-Methylbenzoic
Cinnamic
Furoic
Succinic
Glutaric

Alcohols (a)

Methanol
Ethanol
n-Propanol
i-Propanol
i-Butanol
2-Butanol
i-Pentanol

2-Pentanol
2-Methyl-1-Butanol
3-Methyl-2-Butanol
2-Methyl-2-Butanol
Pent-1-en-3-ol
n-Hexanol
Hex-3-en-1-ol
2-Hepanol

2-ethylhexanol
Benzyl alcohol
Phenylethylanol
p-Hydroxyphenylethanol
Linalool
α-Terpineol
Borneol
Isofenchyl alcohol
Terpin

Esters (a)

Ethyl formate
n-Pentyl formate
i-Pentyl formate
Methyl acetate
Ethyl acetate
n-Propyl acetate
n-Butyl acetate
i-Butyl acetate

Methyl-n-butyrate
Ethyl-n-butyrate
i-Propyl-n-butyrate
n-Hexyl-n-butyrate
trans-2-Hepenyl n-butyrate
Methyl i-butyrate
Ethyl i-butyrate
Ethyl 2-butenoate

Esters (a)

i-Pentyl acetate
n-Hexyl acetate
2-Hexyl acetate
Hex-2-en-1-yl acetate
Benzyl acetate
Ethyl propionate
Methyl n-hexanoate
Ethyl n-hexanoate
i-Propyl n-hexanoate
n-Butyl n-hexanoate
n-Pentyl n-hexanoate
n-Hexyl n-hexanoate
cis-3-Hexenl-yl n-hexanoate
trans-3-Hexen-1-yl n-hexanoate
2-Hexenyl n-hexanoate
Ethyl n-heptanoate
Methyl n-octanoate
Ethyl n-octanoate
Ethyl acetoacetate
Ethyl n-pentanoate
Ethyl i-pentanoate
Methyl-2-methylbutyrate
Ethyl 2-methylbutyrate
Butyl 2-methylbutyrate
n-Pentyl n-octanoate
n-Hexyl-n-octanoate
Hexenyl n-octanoate
Methyl n-decanoate
Ethyl n-decanoate
Ethyl n-dodecanoate
Ethyl benzoate
Methyl trans-cinnamate
Methyl cis-cinnamate
Ethyl trans-cinnamate
Ethyl salicylate
1-Decalactone

Carbonyls (a)

Acetaldehyde
Acetone
Propanal
2-Propenal
n-Butanal
2-Butenal
2-Butanone
2,3, Butanedione
2-Pentenal
2-Pentanone
3-Methyl-2-butanone
Hexanal
2-Hexanal
Cis-3-Hexenal
Heptanol
2-Heptanone
Benzaldehyde
Acetophenone
Furfural
Methylfurfural
2-Acetylfuran

Acetals (a)

1,1-Dimethoxymethane
1,1-Dimethoxyethane
1-Ethoxy-1-methoxyethane
1,1-Diethoxyethane
1-Ethoxy-1-propoxyethane
1,1-Diethoxypropane
1-Butoxy-1-methoxyethane
1-Butoxy-1-ethoxyethane
1,1-Diethoxybutane
1-Methoxy-1-pentoxyethane
1,1-Diethoxypentane
1-Ethoxy-1-pentoxyethane
1-Hexoxy-1-methoxyethane
1,1-Diethoxyhexane
1-Ethoxy-1-hexoxyethane
1-Ethoxy-1-hex-3-enoxy-ethane
1,1-Diethoxypentane
1,1-Dihexoxyethane
1,1-Diethoxyoctane
1,1-Dihexenoxyethane

Hydrocarbons (probably from solvent) (a)

i-Pentane
n-Hexane
Cyclohexane
Methylpentane
Methylcyclopentane
2-Methyl-1-pentene
Benzene
Naphthalene
1-Methylnaphthalene
2-Methylnapthalene

a - Nursten (1970)

b - Mussinan and Walradt (1975)

Although many and perhaps all of the components of strawberry aroma have been identified, few investigators have attempted to determine which compounds contributed significantly to strawberry aroma. As Broderick (1976) pointed out, strawberry has a complex flavor without one or two predominate flavor notes. Assessment of characteristic odor qualities such as aromatic, fruity, floral, fragrant, sweet etc. was difficult for such complex mixtures. A list by Broderick (1976) was compiled indicating the components identified in strawberries and those which are used in synthetic strawberry essence. Recently Dirinck et al. (1977) analyzed strawberry aroma using headspace collection techniques and showed that fresh strawberry flavor was nearly completely composed of volatile organic esters.

Development of Aroma

Enzymic Development of Aroma

Flavor is a complex sensory perception composed of both taste and smell. Aroma is a critical factor in consumer acceptance, identification and assessment of quality in fruits and vegetables. All fruits and vegetables have their own characteristic aroma depending upon the cultivar, maturity and horticultural practices. Development of the characteristic flavor of fruits and vegetables has been demonstrated to be enzyme catlayzed (Reed 1966, Yu et al. 1968a,b, Heatherbell and Wrolstad 1971).

Interest in elucidating the mechanisms and pathways of aroma production was stimulated with Hewitt's (1956) flavorese theory. This concept postulated the regeneration of fresh flavor in processed foods, which contained stable flavor precursors, with addition of flavor forming enzymes. Upon addition of the flavorese enzymes, extracted from the fresh food, non-volatile precursors were converted to their volatile counterparts. Weurman (1961) first used headspace sampling and subsequent GC analysis of volatiles produced from enzyme-substrate reactions from raspberries. A mixture of non-volatile precursors and a crude enzyme extract produced a characteristic raspberry odor. When commercial enzyme preparations were added to the non-volatile precursor

no odor developed (Weurman 1961). Heatherbell and Wrolstad (1971) investigated the regeneration of raw carrot aroma from odorless carrot substrates and flavorese enzymes. These authors found erratic enzymic activity.

Nursten (1970) reviewed the biosynthesis of alcohols, aldehydes, acetals, ketones, acids, lactones, esters, and terpenes in relation to flavors. He concluded by saying

> the biochemical aspect (of flavor research) is probably the most difficult to pursue and, partly because of that, it is the one which most needs further efforts at elucidation.

Seven years later Salunke and Do (1977) reviewed the metabolic pathways and parameters concerning aroma biosynthesis in 20 fruits and vegetables. They pointed out that "information on development and degradation of aroma of fruits and vegetables and their products is still meager at present."

In the seven years since Nursten (1970) first reviewed the biosynthesis of flavor components, little progress has been made. Burchmann and Kolb (1973) investigated the effects of inhibitors on the flavor forming enzyme system from the calyx cone of raspberries by GC. These authors reported strong inhibition by galactose glucono-1,5-lactone, sodium diethyldithiocarbamate and aniline. A sensitivity of α-galactosidase, β-glucosidase metallic enzymes and β-fructosidase was suggested. Amino acids when mixed with

a crude enzyme extract prepared from fresh tomatoes were converted into carbonyl compounds and alcohols (Yu et al., 1968a,b). In banana, the conversion of L-leucine to isoamyl alcohol and isoamyl acetate was found by Myers et al. (1970). Tressl and Drawert (1971) determined that banana slices metabolized 8-^{14}C-octanoic acid via β-oxidation to ketones, alcohols, and fatty acids with simultaneous esterification of octanoic acid and subsequent reduction to octanol. This system was dependent on the climacteric state of the fruit.

Development of Aroma in Strawberries

The development of flavor in strawberries has only been recently studied. Charier-Vadrot (1972) issued an interesting patent for obtaining "aromatic substances" from vegetable tissues. Strawberry shoots were soaked in an aqueous solution of sugar, vitamins and inorganic salts under controlled conditions to obtain a solution that "had the odour and taste of fresh strawberries." Yamashita et al. (1975, 1976a,b, 1977) have published a series of papers on the development of esters in strawberries. They found when various aliphatic alcohols were incubated with a whole strawberry, there was a rapid increase in corresponding esters. Seventy esters were formed when various combinations of acids (C_1 to C_6) were incubated together in

a whole strawberry. When crushed or homogenized (pH 7.0) strawberries were incubated with alcohols, no increase in ester formation was shown (Yamashita et al., 1975). Also significant was that the concentration of esters formed seemed to be dependent on the concentration of corresponding acids contained in the strawberry fruit.

Investigations were further conducted by Yamashita et al. (1976a) on the formation of alcohols, followed by their conversion to esters. In all, seven aldehydes were all reduced to the corresponding alchols
and the alcohols produced were converted to 54 of the corresponding esters. Many of which have been found in strawberry oil. Yamashita et al. (1976a) suggested the existence of an active alcohol dehydrogenase (ADH) system for the conversion of aldehydes to alcohols in strawberries.

Later it was demonstrated strawberry seeds contained at least two ADH; one was nicotinamide adenine dinucleotide (NAD) specific and reacts with ethanol and allyl alcohol, and the other was nicotinamide adenine dinucleotide phosphate (NADP) specific and reacted with benzyl alcohol and geraniol. The maximum stability of the NADP and NAD specific ADHs were both at pH 7.0; although NAD dependent ADH rapidly lost activity at pH greater than 8.5. Both were very labile at pH less than 6.0. Both ADHs were themostable when exposed to temperature less than 45^{o}C for 15 min. (Yamashita et al., 1976b).

Further investigations into the biosynthesis of volatile alcohols and esters from aldehydes in strawberries at different stages of maturity were conducted by Yamashita et al. (1977). Reduction of aldehydes to the corresponding alcohols occured at every stage of maturity but was stimulated with maturity. Biosynthesis of the corresponding esters occured only in partially mature fruit; concomitant with a decrease in corresponding alcohols. Yamashita et al. (1977) reported biosynthesis of volatile acids only occured in partially mature fruit, which parallel very closely the biosynthesis of esters in strawberries. These authors concluded by remarking, "The lack of volatile fatty acid and ester producing enzymes were two factors influencing the absence of ester formation at immature stages."

Evaluation of Volatile Flavors

Subjective Analysis of Volatile Flavors

"Sensory evaluation is concerned with human evaluation and measurement of physical stimuli" (Larmond 1973). Although most studies have investigated the contribution of the sense of taste and smell, other important factors such as appearance, tactile and thermal sensations all interact to give an integrated response. As Harries (1973) pointed out, new methods in assessing the sensory attributes of foods must be developed that account for the total food system.

Unlike the sense of taste, which has been classified into sweet, bitter, sour and salty sensations (Pangborn 1976), the sense of smell could not be classified into a simple system (von Sydow 1971). Experienced judges have been able to distinguish about 20 different levels of intensity (Pangborn 1976). The sense of smell has been shown to be localized in the olfactory epithelium located in the nasal cavity and a compound must be volatile to stimulate the organ. Pangborn (1976) stated

> we do not understand even the most elementary aspects of receptor function. There is general agreement that the initial mechanism of olfactory receptor stimulation depends upon the adsorption of the stimulus molecule to the receptor surface, causing an electrical depolarization of the olfactory cell which initiates nerve impulses.

Adsorption depends upon the molecular structure of the stimulating molecule illiciting the response.

Ammore (1970) postulated that there were a specific number of receptor sites in the olfactory mucosa which accommodated compounds with corresponding shapes and sizes, triggering an impulse which was transulated into an odor quality. Davies (1965) hypothesized that compounds were adsorbed at the receptor site and the associated energy leads to puncture of a membrane to effect a nerve impulse.

Before conducting any type of sensory testing, panelist must be selected and in some instances trained. Martin (1973) reported the selection and amount of training depended upon what was to be evaluated. "Training can go from none, in the case of consumer panelist, to an intensive one year course, in the case of descriptive flavor analysis" (Martin 1973). Panelist have been trained to reduce variability in their individual judgments and to familarize panelists with test procedures. Further information on the selection and training of judges may be obtained from ASTM (1968) and Amerine (1965).

Evaluation of the sensory characteristics of food is subjective in nature requiring human interpretation and measurement. Twenty to twenty-five years ago, subjective studies were viewed as being invalid because they were

insufficiently objective to be scientific. When Stevens (1946) developed the theories of measurement which implied that measurement was simply the assignment of numbers to things, (according to certain rules) subjective assessments gained more respect (Harries 1973). However, many times the researchers have adopted convenient arbitrary scales of 5,7, or 9 points for measuring sensory attributes making comparisons confusing (von Sydow 1971). There is an apparent need for a more systematic approach in the description of aroma and taste qualities.

Objective Analysis of Volatile Flavors

To circumvent problems with sensory analysis design and interpretation, scientist have searched for more objective methods in aroma research. In addition, the food industry has needed a reliable, fast-objective method for analysis of volatile mixtures to better monitor quality of the product. The separation of volatiles by GC has allowed one to obtain a quantative measurement of the aroma under study. Headspace vapor analysis, developed by Miller *et al.* (1972), where the volatiles above the surface of a food were analyzed by GC, is a relatively fast and simple isolation technique. With advent of GC-MS coupled with computer software systems many of the compounds making up aroma have been identified. Establishing which compounds are responsible for a particular attribute has been done

in relatively few cases for complex mixtures. As Jennings (1977) discussed, flavor is due to an intergrated response of many compounds. Syngerism and antagonism between compounds make interpretation of GC data very difficult. Although GC is powerful tool in the objective evaluation of aroma, Wick (1965) reported that GC and human olfaction vary widely in sensitivity to different molecular species. Misleading information such as nonaromatic volatiles may also lead to misinterpretation of results (Williams 1977).

Correlation of Objective and Subjective Flavor Data

The importance in correlating objective and subjective data has been the topic of many papers (Kramer 1969, Presson and von Sydow 1974, and von Sydow 1971). Both Harries (1973) and von Sydow (1971) have suggested, if progress is to be made in correlating objective and sensory data, there must be better physiochemical methods to measure the important aroma and taste substances; better descriptive and systematic sensory methods that incorporate psychophysical methodology to control perception, integration and communication problems; and finally the use of statistical analysis to interpret and correlate instrumental and sensory data. Until recently, the major problem in applying statistical methods to acquire relevant information from GC data has been the complexity of computations involved (Powers 1968). In some foods the characteristic aroma

was made up of a unique compound. For these, simple correlation methods were sufficient. However, chromatograms containing over 100 peaks were not unusual. Patterns of GC data, which have been judged different by a sensory panel, have appeared quite similar. With the application of computers, statistical techniques have been able to ferret out information enabling one to interpret and correlate instrumental and sensory data (Powers 1968).

Application of statistical and psychophysical principles as a means to correlate objective and subjective data are beginning to emerge and yield some good results. Using linear regression analysis on GC data and sensory scores, Fore et al. (1972) found a high correlation between flavor scores and the ratio between 2-methyl propanal and hexanal in peanut butter. Galetto and Bednarczyk (1975) used multiple regression techniques in establishing that the amount of methyl propyl disulfide, methyl propyl trisulfide and dipropyl trisulfide showed a high degree of correlation with onion flavor. Presson (1974) was able to predict the intensities of 15 odor descriptors using multiple regression models containing four GC peak combinations. Powers et al. (1977) used cluster and factor analysis to establish relationships amoung 58 components such as courseness, crispness, color, mouthfeel, GC peak etc. Multivariant regression was then applied to determine the correlation between factors. Lindsay (1977) was

able to correctly classify beer 94% of the time from data on 11 volatiles and 2 mineral salts by using discriminate analysis. Using multiple regression techniques, Carter and Cornell (1977) predicted the flavor of orange juice and obtained coefficients of determinations of .593 to .956 for 10 models.

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ESSENCE RECOVERY FROM STRAWBERRY POMACE

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INTRODUCTION

Flavor is a complex sensory perception. Evaluation of food flavors is difficult because flavor is composed of taste, smell, thermal and tactile sensations. Aroma is a critical factor in consumer acceptance, identification and assessment of quality in fruits and vegetables. Development of the characteristic flavor of fruits and vegetables has been demonstrated to be enzyme catalyzed (Reed 1966, Yu et al. 1968a, Heatherbell and Wrolstad 1971 and Weurman 1961). The metabolic pathways and parameters surrounding aroma biosynthesis were reviewed by Nursten (1970) and Salunke and Do (1977). Both authors commented on the lack of knowledge in this area of flavor research.

Recently Yamashita et al. (1975, 1976a,b) found that when various aliphatic alcohols were incubated with a whole strawberry, there was a rapid increase in corresponding esters. Further work (Yamashita et al. 1976a) showed aldehydes, when incubated with a whole strawberry, were reduced to the corresponding alcohols and the alcohols produced were converted to their corresponding esters. This suggested the presence of an active alcohol dehydrogenase (ADH) system. At least two ADHs were later isolated from strawberry seeds (Yamashita et al. 1976b),

Increasing the strength of natural essences has not been studied extensively. Guadagni et al. (1971a,b) was able to increase the intensity and quality of essence derived from apple peels 2 to 7 times by holding them for 1 to 2 days. Karwowska et al. (1969a) described the production of essence from blackberry pomace. Using pilot plant equipment to produce essences from the pulp juice and pomace of blackberries, they found the essence obtained from the pomace was "most intensive and characteristic of blackberry aroma." Karwowska and Inchas (1969b) later investigated various combinations of the three extracts and found "the richest aroma was obtained by combining the extract from fruit pulp, pomace, and juice."

Since strawberry pomace from juicing operations is not exposed to temperatures in excess of 50^{o}C, it's possible the flavor precursors are not destroyed and enzymes not inactivated. Presently, disposal of the pomace, which retains a great deal of aroma, is costly and wasteful of a valuable raw material. This waste is compounded by the high market value for natural strawberry essence. Therefore, the purpose of this study was to find the optimum conditions for producing a secondary strawberry essence from the pomace.

EXPERIMENTAL

Preparation of Distillates

Collection of essence obtained from strawberry pomace

Juice grade strawberries (Tioga and Hood varieties) and strawberry pomace from the same lot were obtained from a local fruit concentrate processor. The pomace was packed in No. 10 cans as it came from the press chute and placed in insulated carriers containing Dry Ice. After reaching the laboratory the cans were immediately sealed and stored at -40°C until needed.

After thawing at room temperature overnight, 500 g of pomace was placed in a mixing bowl to which 2 l of distilled water were added. The slurry was slowly mixed as the pH was adjusted by addition of 6N hydrochloric acid or 6N sodium hydroxide. The pomace was brought up to temperature in a hot water bath (60°C) prior to placing in a water bath of appropriate incubation temperature. After an appropriate holding time, all samples were further adjusted to pH 3.5 using 6N hydrochloric acid or 6N sodium hydroxide and placed in a 12 l round bottom distillation flask. The pomace slurry was vacuum distilled at 5 cm Hg for 30 min at 40C. Two 60 cm Allihn condensors were used; the first was cooled with cold tap water, the second, which refluxed condensate into the collection flask, was cooled with ice water. A 5.5 fold essence was collected and stored at -5°C until analyzed.

Collection of whole strawberry essence

To simulate the industrial production of natural strawberry essence on a laboratory scale, whole strawberries, obtained from the same lot as the pomace, were juiced in an Oster Automatic Juice Extractor. To the puree, 0.1% Klearzymne 100 (Wallerstein) was added and incubated for one hr in a 46^{o}C water bath. The whole strawberry puree was then distilled using the same equipment and under the same conditions as previously described. A 10 fold essence was collected and stored at -5^{o}C until analyzed. The essence was diluted with distilled water to an equivalent 5.5 fold essence which facilitated the comparison of the essence obtained from the pomace and the whole strawberry essence.

Sensory Evaluation

Experimental design

Two separate series of sensory analyses were conducted. The first investigated the effects of holding temperature (15 to 55°C), holding pH (2.5 to 4.5) and time of incubation (1 to 7 hr) prior to distillation on the intensity and quality of essence produced from strawberry pomace. To investigate these interactions a 5^3 fractional factorial design was used (Cochran and Cox 1957) to determine the combination of holding parameters (time, temperature and pH) when producing the distillation samples. A complete design of three factors at five levels each would require a sensory panel to evaluate 125 samples of recovered essence. Cochran and Cox (1957) described a method to obtain most of the desired information by testing only a fraction of the total number of treatments. Thus a 20 treatment factorial design was used. Distillates were randomly presented to the panel. Multivariate analysis of variance and a central composite rotatable response surface as described by Cochran and Cox (1957) was used to analyze the data.

The second series of sensory analyses investigated the effects of holding the strawberry pomace at pH 4 to 10 and at temperatures of 20 to 50°C. All samples were incubated for 4 hr prior to distillation. Presentation of distillates to the sensory panel was determined by an

incomplete split plot design (Cochran and Cox 1957). Results were subjected to analysis of variance.

Panel evaluation of aroma

For aroma evaluation, 40 ml of strawberry pomace essence, which had been diluted 300% using distilled water, were measured into a stemmed wine glass covered with a 75 mm watch glass. The judges were instructed to swirl the glass, remove the watch glass, sniff, replace the cover and mark the ballot. After waiting one minute the judges proceeded to the next sample.

Fifteen judges were selected on their ability to correctly rank the odor intensity of a dilution series of commercial natural strawberry essence. Using distillates prepared from the pomace, judges were trained to develop an internal aroma intensity scale so as to reduce variability. For the first series of sensory evaluations, seven point scales were used to measure intensity and to measure the quality of the strawberry aroma. Three samples coded with three digit random numbers were presented at each session. Twelve of the original 15 judges were selected for the second series of sensory evaluations. The same ballot was used except a reference point was included on the scale for intensity and a reference sample was presented in addition to the three coded samples per session. The reference sample was an equal mix of four

essences from pomace samples processed at pH 4,6,8, and 10 at 30°C. This represented an average in quality and intensity pomace essence.

GC Analysis

Trapping Procedure

Volatile compounds were trapped using a headspace collection technique as described by Miller et al. (1972). A 250 ml glass bottle, containing a magnetic stirrer, was filled with 25 mls of 5.5 fold strawberry pomace distillate, 25 mls of distilled water and 20 g of anhydrous sodium sulfate. Ethyl esters (C_4 to C_{10}) were added to permit calculation of retention index (Ie) values as described by van Den Dool and Kratz (1963). Ethyl nonoate was used as the internal standard for calculation of normalized peak areas. Prior to collection, the sample was allowed to stand in a 60°C water bath for 10 min to saturate the headspace. The surface of the agitated sample was swept with prepurified N_2 at 12 cc/min for 30 min and the entrained volatile compounds were collected on 100/200 mesh Porapak Q in a 4 in x 0.24 in ID stainless steel precolumn maintained at 55°C. At the conclusion of the entrainment procedure, the precolumn was purged with N_2 for an additional 20 min to remove any excess water. The flow of N_2 was then reversed while maintained at 12 cc/min and the temperature of the

precolumn increased to 135°C. The volatile compounds eluted were condensed in a 10 in x 0.03 in ID U-shaped stainless steel trap packed in Dry Ice. This transfer was completed in 45 min.

GC- Mass spectrometry (MS)

The volatile compounds collected from the various strawberry pomace essence samples in the capillary traps were flashed into a 500 ft x 0.03 in ID stainless steel column coated with SF-96 and 5% Igepal CO 880 using the system described by Scanlan et al. (1968). Helium flow through the injection system and the column was15 cc/min. The column was held at 70°C for 10 min, then programmed at 2°C/min to 160°C. The GC effluent was conducted into the ion-source of a Finnigan Quadrapole Model 1015C electron impact MS and spectra of the eluted compounds obtained. Data was acquired and processed with a System Industries System 250 data system.

RESULTS AND DISCUSSION

First sensory analysis

A very informative method of analysis of the results of a factorial experimental design is a second order response surface (Cochran and Cox 1957). The values assigned by the sensory panel in the present experiment were a function of the parameters time, temperature and pH employed in processing of the pomace prior to distillation assuming that all other factors surrounding the testing conditions were held constant. Two models were developed (Table 1) to predict the intensity and quality of strawberry essence produced from waste pomace. Statistical significance ($p \leq .05$) was found for the intensity model only. The regression coefficient (R^2) value indicated that 51% of the total variation could be explained by the intensity model. All factors (time, temperature and pH) were important elements in the model. Table 1 shows if any of these factors were omitted the R^2 would be decreased more than 50%. However, if temperature effects were removed from the model, the R^2 value indicated that only 10% of the total variation could be explained by the model. Thus, temperature appears to effect the intensity of aroma more than time of incubation or pH.

Although the panel was screened and trained, a large portion of the uncontrolled variation probably came from

Table 1 - Response surface models describing intensity and quality of strawberry pomace when applied to sensory data

RESPONSE	MODEL[a]	R^{2b}		M.D.[d]
Intensity (Significant at .05 level)	$Y = 11.62 - .805\ T - .117\ {}^{o}C - 2.39\ pH - .009\ T^2 - .0009\ {}^{o}C^2 + .048\ pH^2 + .009\ T \times {}^{o}C + .47\ T \times pH + .041\ {}^{o}C \times pH$	Total	.51	
		Drop T	.20[c]	.241
		Drop ^{o}C	.10[c]	
		Drop pH	.21[c]	
Quality (not significant)	$Y = 7.89 - .483\ T + .028\ {}^{o}C - 2.45\ pH + .007\ T^2 - 0.0\ {}^{o}C^2 + .409\ pH^2 + .001\ T \times {}^{o}C + .114\ T \times pH^2 - .012\ {}^{o}C \times pH$			.273

[a]Model for pH 2.5 to 4.5; Temp. 15 to 55^{o}C; 1 to 7 hr. of incubation (T)

[b]R^2 = regression coefficient

[c]Regression coefficient if parameter (T, ^{o}C or pH) is dropped from the model

[d]M.D.= mean difference

the panel itself. This would account for the lack of fit of the model. Kramer (1969) reported "Lack of precision is usually ascribed to the subjective (panel) when correlating to objective measurements." In Table 1, mean difference (M.D.) is the difference between the actual means from the sensory panel and the calculated values computed using the model. Since scoring by the sensory panel was on a seven point scale for both intensity and quality, a M.D. of approximately .2 does not appear appreciably significant.

When values generated by the model were plotted with a three dimensional projection, the changes in intensity and quality of the essence produced from strawberry pomace can be readily seen (Figure 1 and 2). Figure 1 shows a shift in the maximum intensity from pH 2.5 and 15^{o}C to pH 4.5 and 55^{o}C occurs when time of incubation was increased. Although time of incubation had little effect on the quality of essence produced, there was a noticeable increase in quality of strawberry aroma as the holding pH was increased to 4.5 and as holding temperature was decreased (Figure 2). The increase in both quality and intensity of aroma at pH 4.5 was somewhat surprising since the natural pH of strawberries is 3.0 to 3.5. Yamashita et al. (1976b) found the optimum stability of alcohol dehydrogenase in strawberry seeds to be at pH 7.0. Since the microenvironment pH of strawberries may differ greatly from the pH when homogenized,

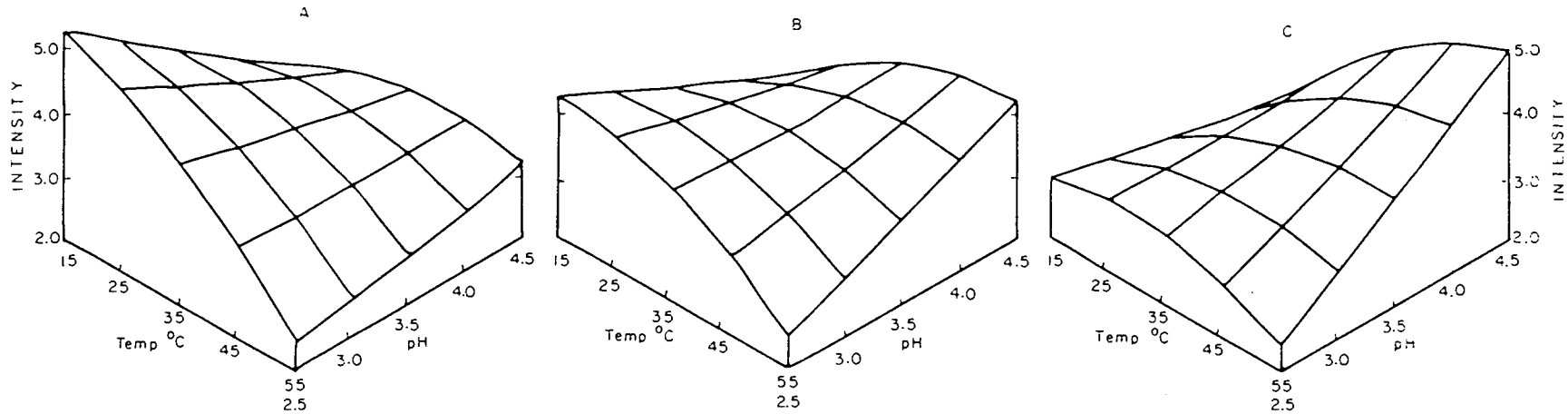
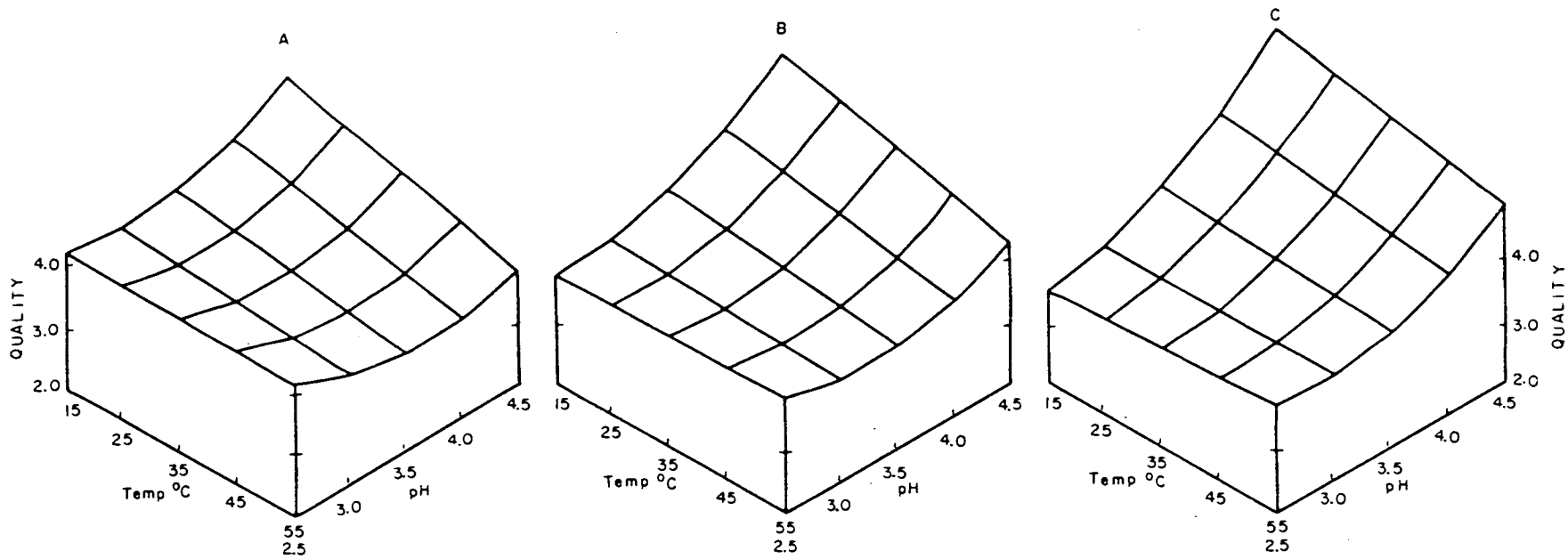


FIGURE 1.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this increase in quality and intensity might be expected. This indicates the presence of an active enzyme system in strawberry pomace which may account for the changes in intensity and quality of essence produced.

Second sensory analysis

Further investigations were conducted to examine the effect of elevated pH on the production of essence from the pomace. Time was not investigated in this second series of experiments since the previous experiment had shown that time of incubation had little effect of quality of aroma. All samples were held at the appropriate temperature and pH for 4 hr. To reduce the panels variability, a reference point was included on the ballot for intensity. The reference sample presented an average intensity and quality strawberry pomace aroma. Results in Figure 3 generally show that at all temperatures as pH was increased, intensity decreased. Judges were able to significantly distinguish between the quality of strawberry aroma at the various pHs and temperatures (Figure 4). An increase, followed by a sudden decrease in quality was obtained for those essences collected from pomace held at 50^{o}C above pH 6. The aroma of these essences contained very little strawberry-like qualities. This could have been due to breakdown products formed due to the high temperature and pH employed. At holding temperatures of 20 to 40^{o}C, quality remained fairly constant, as holding pH was increased. These

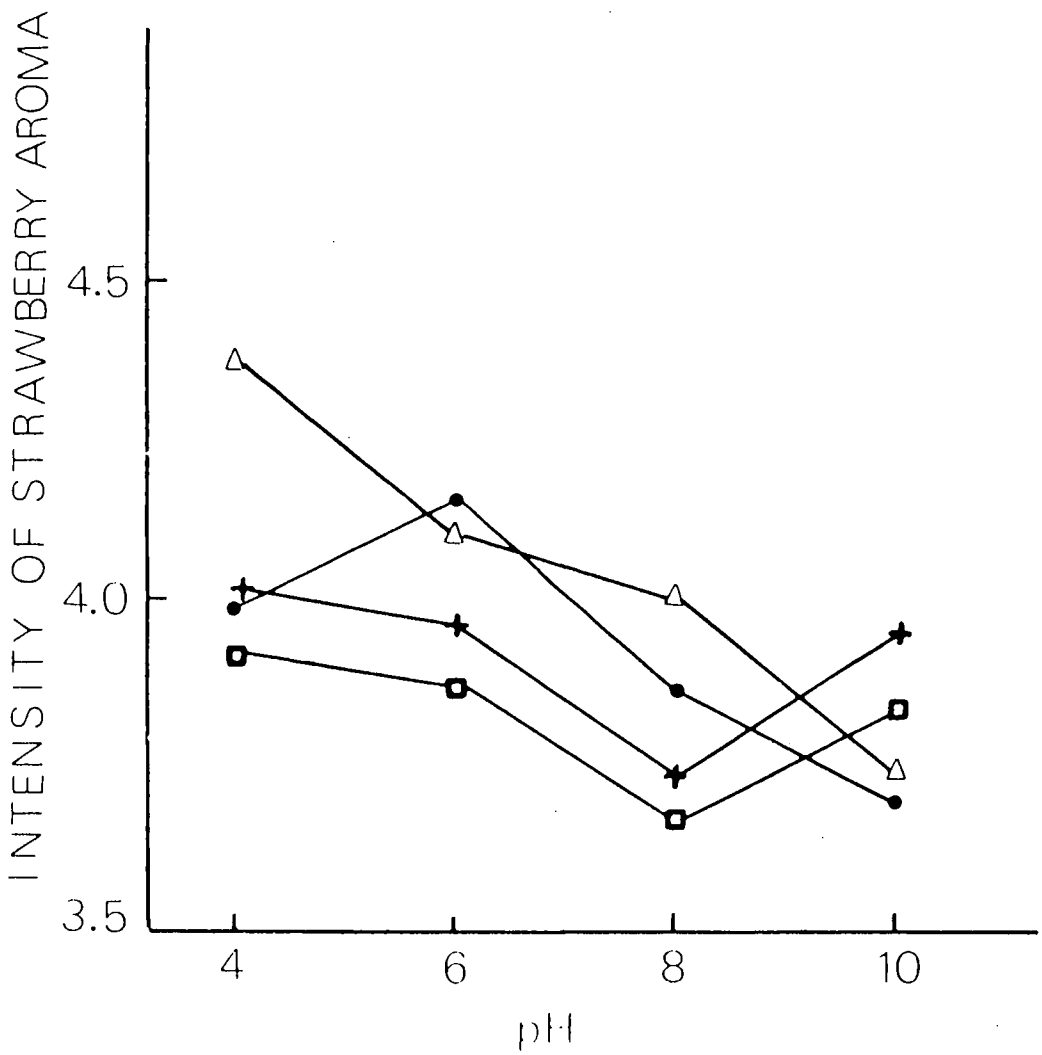

FIGURE 3.

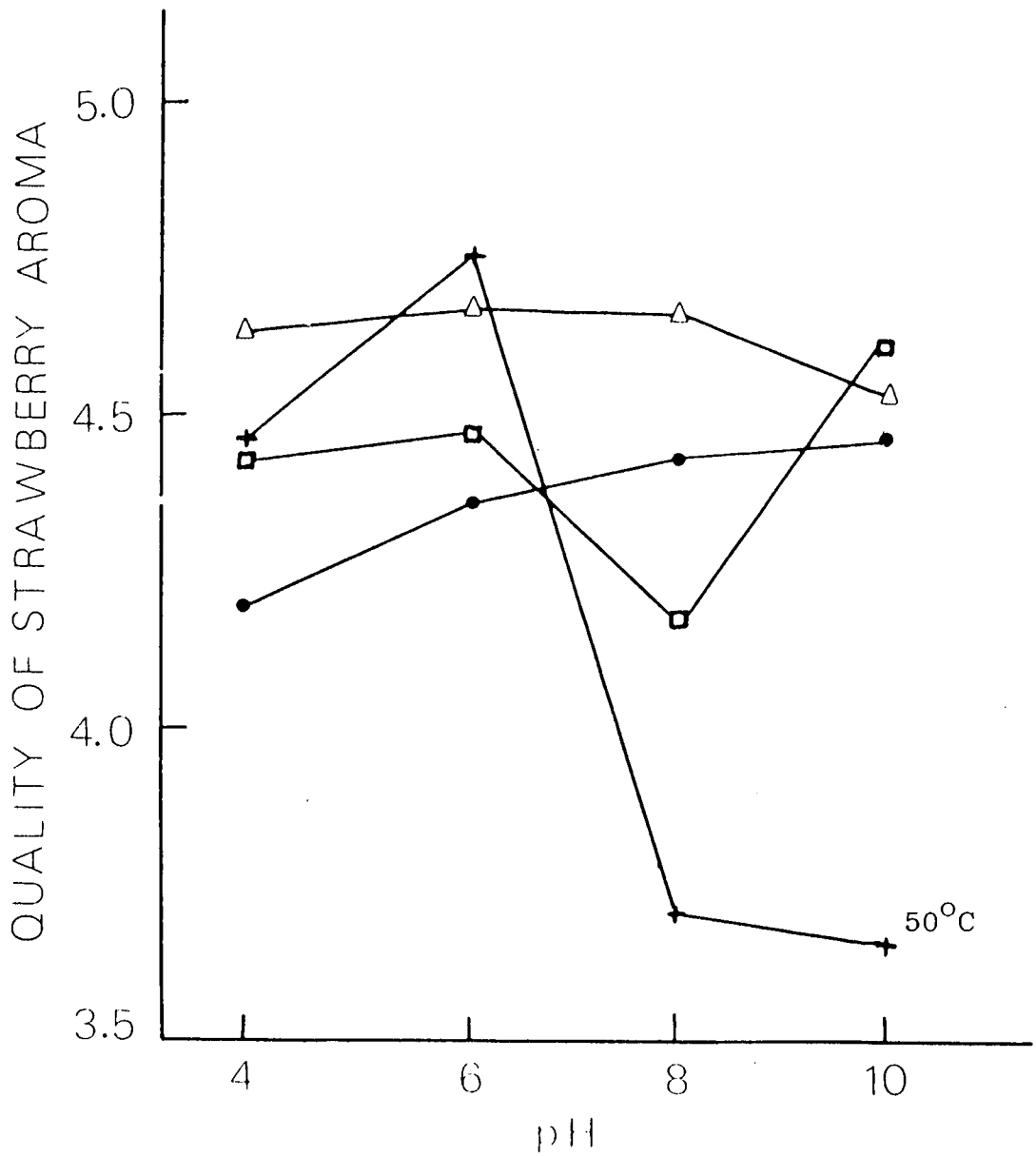

FIGURE 4.

observations in Figure 4 indicate a change in temperature altered quality significantly, suggesting that controlling temperature was important in the utilization of strawberry pomace for the production of essence.

GC analysis

Gas Chromatographic (GC) analysis of whole strawberry essence showed a very complex mixture, predominantely made up of esters. A list of major components is shown in Table 2. These compounds have been previously identified by Teranishi et al. (1963), McFadden et al. (1968), and Tressl et al. (1969). To compare essence from strawberry pomace with essence made from whole strawberries, ratios of major peaks from pomace essence to whole strawberry essence identified in Table 2 were computed. A ratio of 1.0 would indicate the normalized peak area from the pomace essence was equal to the normalized peak area from whole strawberry essence. Samples of similar intensities, but which differed significantly in quality were analyzed (Figure 5 and 6). Judges found the essence represented in Figure 5 was significantly better than that in Figure 6. The pattern of ratios in the two essences differ greatly. As can be seen from the ratios, the distillate judged significantly better (Figure 5) more closely resembles whole strawberry essence. In all low quality

samples examined, there was an increase in n-butyl acetate (26), peak 35, peak 42, methyl hexanoate (45), and α-terpineol (88). These compounds in such large proportions may account for the difference in quality of the two essences, one of which was collected from pomace held at pH 3.5 (Figure 6) the other at pH 4.5 (Figure 5). In all high quality pomace essences examined, 1-pentanol was the only compound whose concentration was consistantly higher than in whole strawberry essence.

None of the chromatograms examined duplicated that of whole strawberry essence. Twenty to 30 new peaks, not found in strawberry essence, were present in chromatograms from the pomace essence. None of these compounds were identified in this study. These additional peaks, in part, could have been due to the filtering aids added to the fruit, such as rice hulls and paper. All pomace essences were abnormally high in benzaldehyde (53), 2-heptenal (50), peak 56, trans-2-hexenyl acetate (59), limonene (61), 2-octenal (64), peak 69, linalool (74), benzyl acetate (83), ethyl cinnamate (119) and peak 125, a sesquiterpene hydrocarbon. This pattern was consistent for all strawberry pomace essences examined, suggesting that orderly enzyme systems acted upon stable aroma precursors in strawberry pomace. The unsaturated aldehydes, found in high concentrations in all pomace essences, might have resulted from action of lipoxidase on

Table 2 - Identification and retention index of major components in whole strawberry essence.

GC Peak No.	Compound	Known Authenitc Standard	Unknown	GC Peak No.	Compound	Known Authentic Standard	Unknown
3	Diacetyl[R]	1.89	1.69	41	Unknown		5.07
4	Ethyl acetate[R]	2.00	2.00	42	Unknown		5.13
6	Isobutanol[M]		2.05	45	Methylhexanoate[R]	5.23	5.23
7	1-Methoxy-1-ethoxy-ethane		2.24	50	2-Heptenal[R]	5.57	5.53
9	Unknown		2.55	53	Benzaldehyde[R]	5.80	5.73
11	Unknown		2.71	56	Unknown		5.90
12	Unknown		2.82	57	Ethyl Hexanoate[R]	6.00	6.00
13	Methyl butyrate[M]		2.89	59	Trans-2-hexenyl acetate	6.15	6.16
14	1,1-Diethoxyethane		3.05	61	Limonene[R]	6.49	6.40
15	3-Pentene-2-one[M]		3.13	64	2-Octenal[R]	6.60	6.59
17	3-Methyl-1-butanol[R]	3.48	3.36	69	Unknown		6.90
22	1-Pentenol[R]	3.88	3.86	71	Ethyl n-heptanoate	7.16	7.00
24	Ethyl butyrate[R]	4.06	4.00	74	Linlool[R]	7.27	7.17
26	n-Butyl acetate[R]	4.12	4.11	75	Methyl n-octanoate[R]		7.30
31	2-Hexenal[M]		4.47	83	Benzyl acetate[M]		7.74
32	Ethyl isovalerate[R]	4.52	4.50	87	Ethyl n-octanoate[R]	8.00	8.00
35	Unknown		4.74	88	α-Terpineol[M]		8.07
37	3-Hexenal[T]	4.87	4.82	110	Ethyl n-decanoate[R]	10.00	10.00
38	1-Hexanol[R]		4.90	119	Ethyl cinnamate[M]		10.35
39	2-Hexanol[T]		4.95	125	Sesquiterpene T		
40	Ethyl valerate[R]	5.00	5.00				

R-MS and retention index identification
M-MS identification
T-tentative MS identification

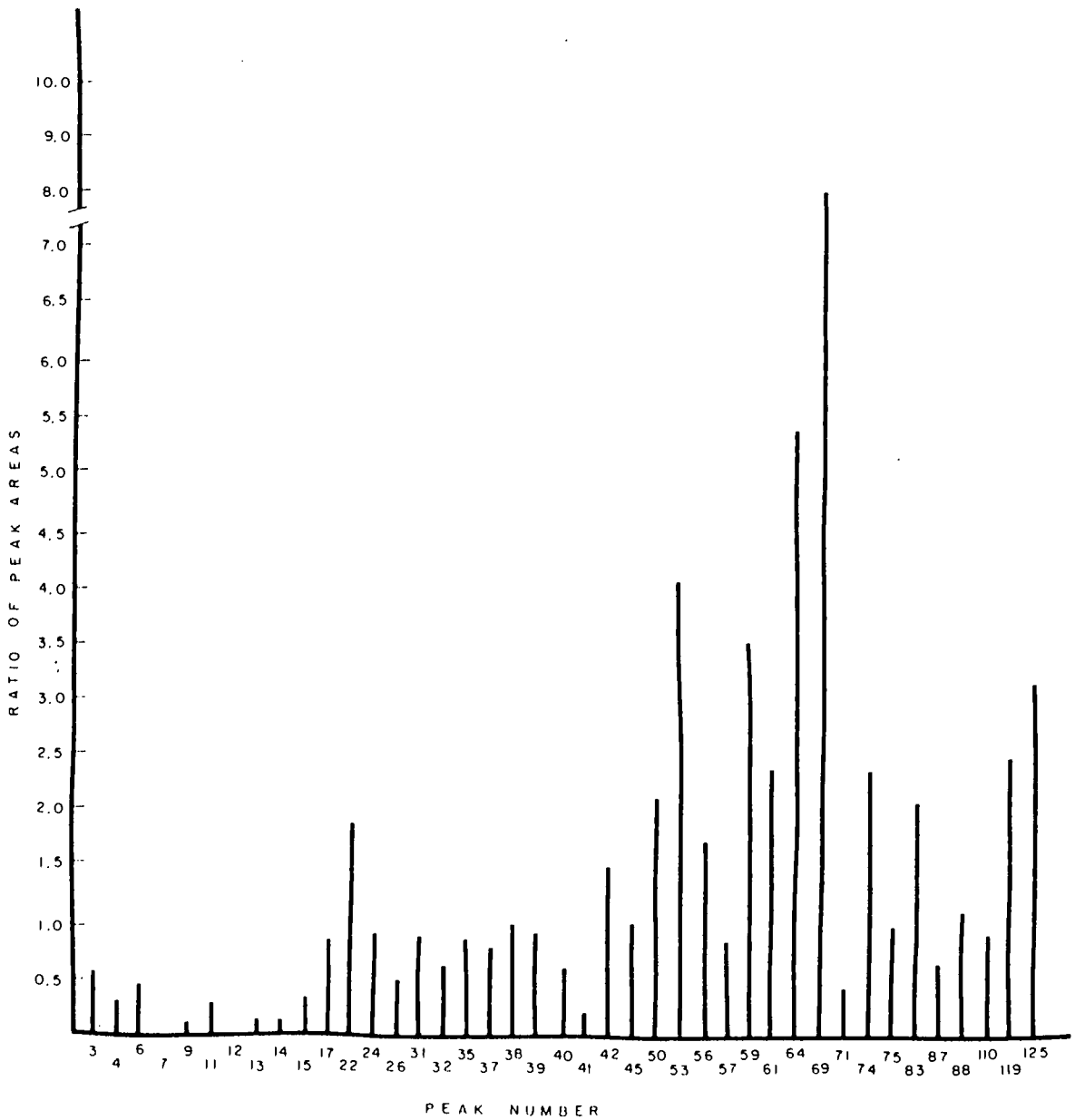
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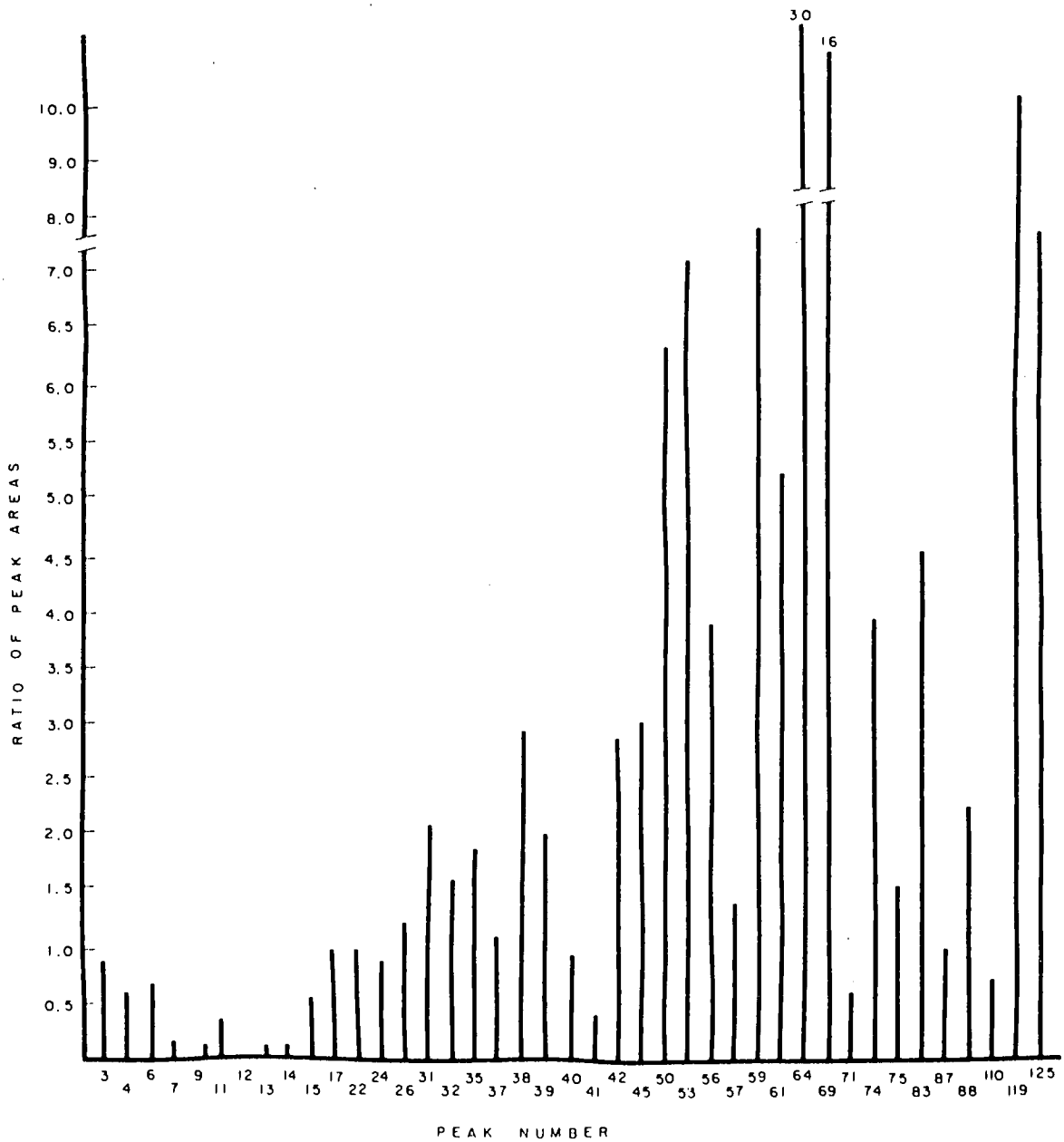
FIGURE 5.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RATIO OF PEAK AREAS
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PEAK NUMBER

FIGURE 6.

unsaturated fatty acids found in strawberry seeds within the pomace. Formation of n-hexanal and trans-2-hexenal from ^{14}C-labeled linoleic and linolenic acids in bananas was reported by Tressl and Drawert (1973). A consistent increase in low molecular weight esters important in strawberry aroma did not occur in any of the pomace essences examined. However, large increases in high molecular weight esters (Methyl hexanoate, trans-2-hexenyl acetate, benzylacetate and ethyl cinnamate) was observed in all pomace essences examined.

CONCLUSION

Whether or not enzymatic action was responsible for the differences in intensity and quality of the strawberry pomace essence is not certain. Judges found statistically significant differences occured in quality of the essence when pH was varied from 4.0 to 10.0 (Figure 4). A study of the GC patterns of volatiles obtained from these essences again indicated major differences. This suggests that an enzymatic process may play a role in production of essence from strawberry pomace.

Although a second order response surface model (Table 1) did not fit the sensory data, multivariate analysis of variance indicated temperature, rather than pH and time, was the most important factor to be controlled in the treatment of strawberry pomace. This is further supported by data in Figure 4 where quality appears to be most influenced by temperature.

From the observations made in this work, duplication of the aroma of whole strawberry essence from strawberry pomace is not possible. However, to obtain the best possible results, the three factors studied (time, temperature and pH) must be controlled. The optimum conditions found in this study were at pH 4.0 and 40^{o}C for 4 hr or longer. An equivalent strength pomace essence processed under optimum conditions, will be slightly less intense and less desirable than that made from whole strawberries.

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