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CONTROL OF OFF-FLAVOR AND ODOR DEVELOPMENT IN PACKAGED FROZEN FISH DURING STORAGE

By

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ABSTRACT

CONTROL OF OFF-FLAVOR AND ODOR DEVELOPMENT IN PACKAGED FROZEN FISH DURING STORAGE

By

Lin-Bin Su

A packaged absorbent to control off-flavor and odor in a packaged product was investigated. Frozen fish (lake trout) was used as the model system.

Volatile compounds were collected on Tenax GC at 21°C from oxidized frozen fish (lake trout). Gas chromatography/mass spectrometry was used to identify the volatile compounds. The major off-flavor compounds found were butanal, cycloheptatriene, pentanal, and hexanal.

The adsorbing capacity of six adsorbents, silica gel, activated carbon, sodium bisulfite, lactose, cellulose, and pectin were investigated by monitoring the concentration of volatile compounds in the headspace of jars containing the above volatiles. Activated carbon had the greatest adsorbing capacity of the adsorbents.

Performance of the adsorbent, activated carbon, to reduce the volatiles from frozen fish packaged in glass jars was studied. The results demonstrated that activated carbon or other adsorbents maybe used to adsorb odors associated with storage of frozen fish.

To my mother, Sheng Huang Su

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INTRODUCTION

Fish is considered an inexpensive sources of animal protein worldwide (Sawyer et al., 1988). Many studies on consumer acceptance of fish indicate that flavor is an important criterion for determination of acceptability (Bremner, 1977,1978; Laslett and Bremner, 1979; Wesson et al., 1979; Connell and Howgate, 1971; Hamilton and Bennett, 1983,1984).

Lipids oxidation is one of the main factors responsible for the formation of off-flavor in fish and other lipid-containing foods. The initiation of lipid oxidation and breakdown of lipid oxidation products can be enzymatic nonenzymatic in nature (Hsieh and Kinsella, 1986). Some of the breakdown products of lipid oxidation, including aldehydes, ketones, and alcohols, contribute to off-flavors in foods (Frankel, 1984). Fish lipids, which contain high amounts of *n*-3 polyunsaturated fatty acids (PUFAs), are very susceptible to lipid oxidation. The oxidation products of *n*-3 PUFAs, particularly aldehydes with an *n*-3 double bond, possess low flavor thresholds (Frankel, 1984). Therefore, once lipid oxidation is initiated, very low concentrations of aldehydes with *n*-3 double bonds can cause distinctive oxidative off-flavors in fish (Hsieh and Kinsella, 1986).

Application of antioxidants to some species of fish before frozen storage does not effectively control the formation of oxidized flavors (Sweet,1973; Deng et al., 1977, 1978; Morris and Dawson, 1979).

This may be due to the uneven distribution and penetration of antioxidants as well as types of antioxidants employed in the system (Josephson et al., 1985).

The products of lipid oxidation may not only be responsible for the development of off-flavors in foods, they may also react with other food constituents such as proteins (Karel,1973). Protein-lipid interactions can occur in some foods and feeds such as frozen and dehydrated fish, fish meal and oilseeds, thus causing some degree of protein damage (Cheftel, 1979).Thus, using the adsorbents to reduce the off-flavor volatiles in the headspace of frozen fish could eliminate or reduce offensive odors. The palatability of frozen fish would be improved and the protein quality of the frozen fish could be assured by preventing further reactions induced by presence of the off-flavor volatiles.

The objectives of this study were:

1. To identify the off-flavor volatiles in the headspace of frozen fish using GC/MS analysis.
2. To evaluate the capacity of six adsorbents to reduce off-flavor volatiles in sample containers.
3. To select the most promising adsorbent and to determine its performance in a package containing frozen fish.

LITERATURE REVIEW

Lipids in Fish

Fish lipids are characterized as highly unsaturated lipids due to their high concentrations of polyunsaturated fatty acids (PUFAs), such as 20:5 *n*-3 (5,8,11,14,17-eicosapentaenoic acid) and 22:6 *n*-3 (4,7,10,13,16,19-docosahexaenoic acid). PUFAs containing 2 or more double bonds are susceptible to oxidation (Olcott, 1962).

The composition of fish lipids is different from most other naturally occurring oils and fats in :(1) possessing larger quantities of fatty acids with chain lengths exceeding 18 carbons, (2) containing a much greater proportion of highly unsaturated fatty acids, and (3) having polyunsaturaturates, primarily at the ω-3 rather than ω-6 position (Flick et al., 1992). In addition, the amounts and character of the fatty acids vary with the different organs and parts of the fish (Flick et al., 1992).

Lipid Oxidation in Fish

Lipid oxidation is one of the most important factors responsible for quality loss of fish during refrigerated and frozen storage. For example, lipid oxidation causes changes in the flavor, color, and texture of fish (Khayat and Schwall, 1983). Lipid oxidation in fish may be nonenzymatic, such as autoxidation and photosensitized

oxidation, or initiated by enzymes, such as lipoxygenases, peroxidases and microsomal enzymes (Hsieh and Kinsella, 1986).

Lipid oxidation involves the chemical breakdown of fatty acids in the presence of oxygen. It is a rather complex process, where unsaturated fatty acids react with molecular oxygen via a free radical chain mechanism to form fatty acyl hydroperoxides, generally called peroxides. They are primary products of the oxidation process (Gray, 1978). The hydroperoxides formed are very unstable and can breakdown to form free radicals, which in turn can accelerate the rate of lipid oxidation (Flick et al., 1992). The hydroperoxides may also undergo carbon-carbon cleavage to produce volatile compounds such as aldehydes, ketones and hydrocarbons. The free radical chain reaction is self-generating. It continues even at freezing temperatures, especially in fish with high fat content, and is not especially affected by antioxidants (Flick et al., 1992).

Deterioration of Fish Lipids during Cold Storage

Low temperature storage can slow down enzyme activity and inhibit microbial growth in fish. The loss of quality of fish prior to freezing is due to one or all of several factors, such as physical damage, bacterial spoilage or autolytic breakdown of the flesh adjacent to the abdominal cavity. The main factor limiting the frozen storage life of herring was the hydrolysis of lipids during frozen storage (Bilinski et al., 1978). The hydrolysis of lipids in herring during chilled or frozen storage lead to the formation of free fatty acids which are present due to enzymatic degradation of triglycerides and phospholipids (Bosund and Ganrot, 1969).

Many investigators have studied changes in the lipid fractions in fish muscle during frozen storage and found that the increases in free fatty acid content in fish during cold storage are primarily due to the hydrolysis of the phospholipids which are catalyzed by phospholipases in thall et al. 1950; Grieg, 1968a,b). Bligh and Scott (1966) found an increase of the free fatty acid content from 5 to 325 mg/100g flesh in cod muscle at -12°C for nine months and reported that this was due to the hydrolysis of phosphatidylethanolamine(PE) and phosphatidylcholine(PC). They also found that PE hydrolysis ceased after storage for 4 months, whereas PC hydrolysis continued at a slower rate. Furthermore, Bosund and Ganrot (1969) studied the formation of free fatty acids in herring and found that the increase was primarily due to hydrolysis of lecithin, cephalin and to a lesser extent other triglycerides. The free fatty acids formed in the dark and white muscle as a result of phospholipid hydrolysis were 45% and 75% of the free fatty acid formed in the dark and white muscle respectively (Bosund and Ganrot, 1969a). In addition, the formation of glyceryl-phosphorycholine and choline in cold stored rainbow trout muscle is evidence for enzymatic hydrolysis of phospholipids in fish during cold storage (Jonas and Bilinski, 1967). Nair et al. (1979) and Mai and Kinsella (1979) also demonstrated that the formation of free fatty acids in frozen fish is associated with hydrolysis of phospholipids. They found no significant changes in triglycerides and unsaponifiable matter in fish during frozen storage at -18°C.

Free fatty acids can cause denaturation of proteins in fish flesh (Lovern et al., 1959; Olley and Lovern, 1960; Bligh, 1961; Jonas and Tomlinson, 1962). In addition, the toughened texture, poor flavor,

and unappealing odor in poorly stored frozen seafoods have been attributed to the binding of oxidized unsaturated lipids to proteins, a process by which insoluble lipid-protein complexes are formed (Khayat and Schwall, 1983).

Off-flavors Developments in Fish due to Lipid Oxidation

The quality and shelflife of fish are greatly influenced by the development of oxidative flavors (Josephson et al., 1987). Josephson and Lindsay (1986) indicated that certain volatile compounds responsible for the aroma of freshly harvested fish are the result of the degradation of site-specific (i.e. enzymically formed) hydroperoxides. These enzyme-mediated degradations are expressed in the formation of a variety of alcohols and carbonyls which are present in the highest concentrations in the skin and slime fractions of fish (Josephson et al., 1987).

Volatile aldehydes have long been believed to contribute strongly to the characteristic aroma of oxidized fish lipids (Lea, 1953; Yu et al., 1961; Aitken and Connell, 1979; Ikeda, 1980). These oxidized flavors have been frequently described as rancid, cod-liver-oil-like, and painty (Yu et al., 1961). McGill et al. (1977) reported that 4-heptenal, and 2,4-heptadienal, increase during cold storage of cod. These compounds apparently result from the oxidation of *n*-3 polyunsaturated fatty acids which are released from phopholipids (Ross and Love, 1979).

It has been reported that aldehydes with an *n*-3 double bond are the critical components of oxidative off-flavors because of their low flavor threshold values (Frankel, 1984). Fish lipids, which are

rich in *n*-3 polyunsaturated fatty acids (PUFA) are very susceptive to lipid oxidation. Once lipid oxidation is initiated, very low concentrations of aldehydes with *n*-3 double bonds can cause distinctive oxidative off-flavors (Hsieh and Kinsella, 1986).

Badings (1973) and Ke et al. (1975) concluded that the odor of cold-stored fish could not be attributed to an individual compound, but instead must be caused by a complex mixture of carbonyl compounds. This view has been supported by the observations of Swoboda and Peers (1977) who found that solutions containing both C_7 and C_{10} - 2,4-dienals possessed fishy odors, and that individual compounds eluting through an effluent splitter after gas chromatograph did not exhibit fishy aromas.

Measurement of Oxidative Off-flavors in Fish

Headspace analysis using gas chromatography (GC) is often used to determine flavor compounds of foods. In this procedure, gaseous volatiles associated with food are determined by GC. In some instances, the concentrations of volatiles in the headspace are too low to be detected directly by GC. Therefore, collection and concentration of these volatile substances are necessary prior to final GC analysis (Buckholz et al., 1980). In order to concentrate trace aroma volatiles present in headspace gases over foods, beverages, and environmental samples, adsorption of these volatiles onto a variety of porous solids has become an established procedure for preparing samples for analysis by gas chromatography and mass spectrometry (Nunez et al., 1984; McNally and Grob, 1985a, b). Novotny et al. (1974a) used an adsorption precolumn packed with

porous polymers for high resolution GC analysis of the volatile constituents of body fluids. Murray (1977) used a technique for concentration of headspace, airborne, and aqueous volatiles on a porous polymer precolumn. Recently, Tenax GC (2,6-diphenylparaphenylene oxide polymer) has emerged as a widely used porous polymer for these applications. According to Butler and Burke (1976), Tenax-GC works well for high boiling components due to its high thermal stability and low retention volume. This stability assures no bleed on GC columns during analysis and complete regeneration of the porous polymer by heating the column to 260^{o}C under a purge of helium gas. The polymer containing adsorbed headspace volatiles is stable for up to 5 days at 0^{o}C with no decomposition of adsorbed volatiles (Butler and Burke, 1976). Therefore, concentration of headspace volatiles can be achieved by repeated adsorption of volatiles on the same trap (Mussinan, 1978). Some applications of the general properties of adsorptive techniques in the literatures include retention volumes for different components (Kuo et al., 1977; Krost et al., 1982; Kawata et al., 1982) and the effects of sampling times and purging rates on retention of volatiles on Tenax GC (Buckholz et al., 1980).

While earlier studies have employed a variety of collection devices and procedures, most have utilized thermal desorption in association with transfer techniques for recovery of volatiles for subsequent GC analysis (Bertsch et al., 1975; Murray, 1977; Buckholz et al., 1980; Nitz and Julich, 1984).Simple Tenax-GC collection procedures using readily available materials, e.g. ethyl ether, to elute

adsorbed volatiles have been developed and evaluated (Olafsdottir et al., 1985).

Analytical Methods to Determine Level and Identity of Volatile Compounds

Food flavor consists of many volatile compounds in very small quantities. The aroma of a food is dependent on the vapor pressure of the volatile compounds and interaction of these volatile compounds with the macromolecules which constitute the food matrix. Therefore, the isolation and identification of volatile compounds which contribute to the flavor of a food are difficult (Drumm, 1988).

Isolation of volatile compounds from the food matrix is a critical step in their identification. Two approaches have been directed in the analysis of volatile compounds isolated from foods. First is total volatile analysis which determines the qualitative and quantitative composition of all volatile compounds that can be isolated from a food. Distillation and extraction are the primary methods used in total volatile analysis (Sugisawa, 1981). Direct vapor analysis is the second technique used to sample the actual aroma in equilibrium with the food and is representative of the aroma smelled (Weurman, 1969).

Isolation of Volatile Compounds

Isolation of volatile compounds from a fatty sample is one of the most difficult analytic procedures. Direct extraction with an organic solvent is practically impossible because most organic

solvents dissolve fatty materials. Thus, steam distillation is the most common method of separating organic compounds from fatty materials. A simultaneous steam distillation-solvent extraction apparatus was later applied to analysis of volatiles from fat by several researchers (Buttery et al., 1977; Ohnishi and Shibamoto, 1984). In contrast to steam distillation methods, the extracting solvent is continuously recycled during the distillation procedure. Therefore, only small quantities of solvent are needed for the extraction of volatile compounds from large quantities of food. In this method, the volatiles are extracted from the condensed steam distillate by a low-boiling water immiscible organic solvent in a common condensation region. The aqueous and organic phases are continually returned to their respective flasks during the procedure (Likens and Nickerson, 1964). The Likens-Nickerson procedure and other methods which involve distillation at atmospheric pressure, should be avoided when characterizing the flavor constituents of fresh, uncooked food products. The application of heat results in the formation of artifacts which are typical of the processed, rather than the raw product (Weurman, 1969). Extraction of flavor compounds using Likens-Nickerson apparatus may also result in artifact formation via lipid oxidation (McGill and Hardy, 1977; Lovegren et al., 1979).

The development of porous polymer traps such as Tenax GC (p-2,6-diphenyl-p-phenylene oxide) for trapping volatiles has allowed successful collection of volatiles. Purge and trap techniques are widely employed. Basically, the sample is heated in a glass tube through which a stream of carrier gas flows to carry the volatiles

onto a Tenax trap. The volatiles are then thermally desorbed from the Tenax and swept directly into the GC injection port. This method has been successfully used by Shahidi et al. (1987) for the hexanal analysis of cooked ground pork and by Selke and Frankel (1987) for the analysis of soybean oil volatiles. However, one weakness of this method is that some materials are heat labile. This may result in some formation of a more complex mixture at the temperatures (120°C or higher) used to drive the volatiles onto the GC column (Waltking and Goetz, 1983).

A third method involves collection of volatiles on Tenax GC traps by either a purge and trap method (Olafsdottir et al., 1985; Barbut et al., 1985) or a vacuum extraction procedure (Vercellotti et al., 1987). Following collection, the volatiles are eluted with diethyl ether and injected onto a GC column to resolve the volatiles. Using this method, Barbut et al. (1985) identified hexanal as the volatile compound primarily responsible for the aroma of oxidizing turkey sausage.

Identification of Volatile Compounds

Techniques that combine gas chromatography(GC) and mass spectrometry(MS) have found wide application in flavor research (Maarse, 1991). The GC-MS combination is by far the most popular technique for the identification of volatile compounds in foods and beverages. Many reviews have been published on this subject. Crawford et al. (1976) used GC-MS techniques to identify volatiles from extracted commercial tuna oil. There were 64 compounds identified in the oxygenated fraction and 62 in the hydrocarbon

fraction. Volatile compounds collected on Tenax GC at room temperature (21°C) from direct headspace purging of fresh uncooked whitefish (*Coregonus clupeaformis*) were separated by gas chromatography and identified by mass spectrometry (Josephson et al., 1983). Two distinct families of compounds associated with cucumber and melon fruits, and mushrooms were identified as principal contributors to fresh whitefish aroma. Josephson et al. (1984) used the same techniques to identify the enzymically derived volatile aroma compounds in saltwater and freshwater fish. Volatile compounds collected on Tenax GC at 21°C from oxidized whitefish were separated by fused silica capillary gas chromatography and identified by mass spectrometry (Josephson, 1984). Galt and MacLeod (1984) used a modified headspace sampling procedure involving adsorption of the headspace vapors onto Tenax GC. Rapid heat desorption transferred the volatiles directly into a gas chromatography column for separation. By use of combined gas chromatography/mass spectrometry, 67 identifications were made, including 8 compounds tentatively identified for the first time in cooked beef aroma.

Prevention of Off-flavors Development in Fish during Storage

Although off-flavor development has been recognized as a major problem in frozen fish, several methods can be employed to control or minimize this problem. For example, low temperature storage, application of antioxidant, and packaging technique have been researched and can be used quite successfully.

In this section, the effect of storage temperature on the quality of frozen fish, antioxidant activity and application to fish products, and the packaging requirements for frozen fish will be reviewed. The role of adsorbents, in control of off-flavor development in frozen fish during storage, will be described.

Effect of Storage Temperature on the Quality of Frozen Fish

The effect of storage temperature on the quality of frozen fish has been intensively studied by many researchers (Young, 1950; Dyer and Morton, 1956; Peters and McLane, 1959). Their results indicate that product shelf life is extended by lowering the storage temperature to -18°C or below. Temperature fluctuation of -10 to -4°C has been reported by Lentz and Rooke (1960) in frozen fish shipped by road in refrigerated trailers. A survey conducted by Lane (1966) indicated that some frozen seafoods in retailers' freezer cabinets reached -4°C mainly due to the overloading of products in the cabinets. Dyer (1959) pointed out that storage temperature fluctuation above -18°C caused quality deterioration and shortened the storage life of cod fillets. Palmateer et al. (1960) also found an increase in degree of lipid oxidation caused by temperature fluctuation in frozen rockfish fillets.

The Activity and Application of Antioxidant in Fish Products

Freezing technology provides consumers with products that have reasonably good acceptance. However, rancid flavor has been considered the major factor responsible for the quality loss in frozen mullet fillets (Saenz and Dubrow, 1959; Beaumariage et al., 1969).

Attempts have been made to stabilize seafood products with antioxidants such as ascorbic acid (Andersson and Danielson, 1961), tocopherols (Brown et al., 1957), and butylated hydroxyanisole-butylated hydroxytoluene (BHA-BHT) combinations (Yu et al., 1969). However, none of these antioxidant treatments have been found to be effective in preventing the development of oxidative rancidity. Stuckey (1968) reported that application of phenolic antioxidants in frozen fish fillets is not effective in retarding oxidative rancidity. This may be attributed to either inadequate distribution of these water-insoluble antioxidants in the fish samples or because the antioxidants may simply have lacked sufficient potency in the particular application (Sweet, 1973).

However, Sweet (1973) found that tertiary butylhydroquinone (TBHQ) has significant antioxidant effects in salmon. Due to the high metal ion content in marine fish, he also evaluated the effectiveness of chelating agents (citric acid and EDTA) on stability of fish lipids. He reported that EDTA is a more effective chelator than citric acid and is also effective even when no phenolic antioxidant is present. However, the most potent inhibitors are combinations of EDTA or citric acid and TBHQ or BHA. The most effective stabilizer combination in salmon is TBHQ and citric acid (Sweet, 1973).

A number of studies have also been conducted to evaluate the effectiveness of ascorbic acid as antioxidant in fish products (Bauernfeind et al. 1951; Marcuse, 1954; Lilzemark, 1964; Greig, 1967; Liu and Watts, 1970; Love and Pearson, 1974; Deng et al., 1977).The results of these studies vary considerably and are inconclusive. Some reported that addition of ascorbic acid did not

improve stability of lipids and actually acted as a prooxidant. The nature of the flesh products in which ascorbic acid is incorporated may have direct bearing on the results because of their different biochemical components and other inherent environmental effects (Deng et al., 1978).

Phosphates are also widely used to assist in the preservation of minced fish blocks although oxidative rancidity is controlled more effectively with BHA, BHT and other phenolic antioxidants. Excessive use of phosphates and salt in fish can lead to rubbery texture during prolonged storage (Kirk and Sawyer, 1991).

Packaging Requirements for Frozen Fish

The materials and styles of packaging used for frozen fish (products) are numerous and vary from flexible plastic polybag for retail sale products to waxed paper or polyethylene-lined board for industrial blocks of fillets. Their efficacy depends on their degree of imperviousness to water vapor at low temperatures and the degree to which the product is closely and completely enclosed (Connell, 1990).

A great deal of fish is sold in the frozen food section of supermarket. The factors affecting the quality of frozen fish are : (1) moisture loss-dehydration, (2) oxidation, (3) rancidity, (4) change in odor and flavor, (5) loss of volatile flavors, (6) enzymatic activity, and (7) loss of vitamins (Sacharow and Griffin, 1980). Proper packaging for frozen fish is essential, because efficient packaging will help to offset the detrimental effects of oxygen and of desiccation (Sacharow and Griffin, 1980). Therefore, the package must be

impermeable to water vapor. In addition, there should be no airspace between the product and the package; otherwise the product will undergo dehydration at the site of any airspace, regardless of the impermeability of the packaging material to water vapor. Furthermore, the package should also be impermeable to oxygen in order to prevent rancidity.

Gas impermeable packaging for fresh fish should be used only in systems where there is no chance that the product will be exposed to temperature above 4.4°C (40°F). The reason for this concern lies in the danger of an outgrowth of *Clostridium botulinum*, a genus of relatively ubiquitous bacteria. These bacteria grow in the absence of oxygen and produce a toxin that cause botulism, a potentially fatal disease. Since quality assurance requires a commitment to keeping the product at 0°C (32°F), there is no reservation in a quality assurance program about recommending the use of gas-impermeable packaging (Gorga, 1988).

Wax-coated cartons were the first retail packages used for frozen fish. Problems were encountered with wax flaking off and with dehydration through the score lines on the package. The arrival of polyethylene-coated paperboard offered good flexibility and better moisture protection. Although this was an improvement over wax cartons, problems still remained in heat sealing, ink adhesion and delamination when in contact with fish juices.

Most cartons are coated with petroleum wax-resin blends (hot melts). If the hot melt is used on the inside of the carton, a high gloss is left on the product surface. This offers pleasing aesthetics. The hot

melts are easily heat-sealed. Cartons may incorporate tear strip openings or other convenience features.

The most popular package for fresh refrigerated fish consists of a shallow tray and transparent film overwrap. The tray may be fabricated from molded pulp, foam polystyrene or clear polystyrene. Foam and clear polystyrene require the use of absorbent blotters. The overwrap is usually polyvinyl chloride (Sacharow and Griffin, 1980).

Packaging in oxygen-impermeable flexible films offers practical extension of storage life but the oxygen in the package must be removed before storage either by evacuating the space between film and product or by replacing the air with an inert gas like nitrogen. Vacuum packaging is found to be more practical, though it is only applied to expensive, products, like shrimp and trout. The most suitable material must maintain an excellent oxygen barrier. Laminated structures such as cellophane-aluminum foil-polyethylene or polyester-PVDC-polyethylene are desirable materials. Rancidity formation decreases drastically in vacuum packs employing low oxygen permeable laminates. Overall shelf-life is extended by almost 100%.

Since vacuum packaging does not remove all oxygen from the fish, lipid oxidation still occurs, but at a much slower rate. Proper freezing storage is essential for all packaged fish, whether it be vacuum-packed or overwrapped with a plastic film(Sacharow and Griffin,1980).

Adsorbents Having the Capability of Adsorbing Off-flavors

In recent years, adsorption polymers have been used for collection, and concentration, with subsequent gas chromatography (GC) analyses in a wide variety of applications (Buckholz, Jr. et al., 1980). Novotny et al. (1974a) used a porous polymer adsorption precolumn for high-resolution GC analysis of the volatile constituents of body fluids. Murray (1977) described a technique for concentrating headspace, airborne, and aqueous volatiles on a porous polymer precolumn, and Zeldes and Horton (1978) used Tenax GC adsorption polymer to trap volatiles in cigarette smoke. The use of adsorption polymers has become widespread in the testing of air pollutants (Murray, 1977). Buckholz, Jr. et al. (1980) reported the application and characteristics of polymer adsorption method used to analyze flavor volatiles from peanuts. Josephson et al. (1984) used the adsorption polymer to identify volatile aroma compounds from oxidized frozen whitefish. Galt and MacLeod (1984) optimized the technique for isolating genuine cooked beef aroma volatiles, using a modified headspace sampling procedure involving adsorption of the headspace vapors onto Tenax GC.

In this section, the adsorption capability of six adsorbents (silica gel, activated carbon, sodium bisulfite, cellulose, pectin, and lactose) are reviewed. These adsorbents will be discussed with regards to their ability to adsorb volatile organics.

Silica Gel

Silica gel is a partially dehydrated form of polymeric colloidal silicic acid. The chemical composition can be expressed as SiO_2-nH_2O.

The water content, which is present mainly in the form of chemically bound hydroxyl groups, amounts to about 5% (wt./.wt.) typically. It is a microporous structure in which the pore size is determined mainly by the size of the original micro particles. Bond formation between adjacent particles occurs with elimination of water between neighboring hydroxyl groups and the final structure is therefore physically robust (Ruthven, 1984).

The presence of hydroxyl groups imparts a degree of polarity to the surface so that molecules such as water, alcohols, phenols, amines (which can form hydrogen bonds) and unsaturated hydrocarbons (which can form p-complexes) are adsorbed in preference to nonpolar molecules such as saturated hydrocarbons (Ruthven, 1984). Because of its selectivity for aromatics, silica gel was used as the adsorbent in the Arosorb process for separation of aromatics from paraffins and naphthenes.

Shantha and Ackman (1991) used silica gel to adsorb longer-chain polyunsaturated fatty acids from food and marine lipids for the concentration of these longer-chain polyunsaturated fatty acids. This method did permit a rapid concentration of polyunsaturates from most fish oil and animal lipid samples. It also could be useful for rapid screening of various oils and/or lipids from mixed food products, and especially those containing fish products, for the presence of the C_{20} and C_{22} "omega-3" polyunsaturated fatty acids (Ackman and Ratnayake, 1989b). The information could eventually help in predicting the oxidative stability of food fats and thus their potential for off-flavor development (Peers and Coxon, 1986).

Activated Carbon

Activated carbon is normally made by thermal decomposition of carbonaceous material followed by activation with steam or carbon dioxide at elevated temperature (700-1100°C) (Ponec et al., 1974). The activation process involves the removal of carbonization products formed during pyrolysis, thereby opening the pores of activated carbon (Ruthven, 1984).

The structure of activated carbon consists of elementary microcrystallites of graphite, these microcrystallites are stacked together in random orientation and it is the spaces between the crystals which form the micropores (Ruthven, 1984).

The surface of carbon is essentially nonpolar although a slight polarity may arise from surface oxidation. As a result, carbon adsorbents tend to be hydrophobic and organophilic. They are therefore widely used for the adsorption of organics in decolorizing sugar, water purification, and solvent recovery systems as well as for the adsorption of gasoline vapors in automobiles, and as a general purpose adsorbent in range hoods and other air purification systems (Ruthven, 1984).

In order to recover volatile components from cigarettes during tobacco-roasting, Matsukura et al. (1984) used activated carbon to adsorb volatiles from roasted tobacco and desorptive recovery by ether extraction.

Sakaki et al. (1984) used activated carbon to collect the headspce volatiles swept by helium from cut tobacco and analyzed the volatiles by gas chromatography after desorption by extracting the activated carbon with dichloromethane.

Peanut protein products contain phenolic compounds (Conkerton et al., 1983; Daigle et al., 1983) which have been implicated in off-flavor and color defects (Maga, 1978; Sosulski, 1979). Ion exchange and activated carbon treatments have been successfully used to remove phytic acid and phenolic compounds from soy protein extracts (Brooks and Morr, 1982; How and Morr, 1982). Seo and Morr (1985) used these procedures to remove phytate and phenolics from laboratory and commercial peanut proteins.

Tausig and Drake (1959) incorporated packets of active carbon within containers to adsorb objectionable volatiles of radiation-sterilized beef during storage. A panel of experts judged the intensity of irradiation flavor of cooked beef to be significantly lowered by this carbon treatment (Tausig and Drake, 1959).

A study was made of the use of activated carbon with irradiated cooked pork, chicken, and beef during storage by Gernon et al. (1961). The meat products were evaluated by a consumer-type panel for the combined characteristics of flavor, appearance, and texture, rather than just the single parameter of irradiation flavor.

Toro-Vazquez et al. (1991) evaluated an adsorption system using activated carbon, and the effect of adsorption time, temperature, and concentration of H_2O in the adsorption system on the chemical characteristics of squash (Cucurbita moschata) seed oil.

An ultrafiltration-activated carbon system has been developed to renovate contact refrigeration brines, such as used on fishing vessels, before reuse and to salvage usable byproducts (Welsh and Zall, 1984).

Sodium Bisulfite

Aqueous sodium bisulfite has been evaluated as an adsorbent for volatile chemicals in the headspace above heated pork fat (Yasuhara and Shibamoto, 1989). The major volatile compounds produced from the heated pork fat were hexanal, heptanal, and pentanal (Yasuhara and Shibamoto, 1989).

Volatile carbonyls have been found to be important in fresh fish aroma (Berra et al., 1982; Josephson et al., 1983a). Thus, the aromas of both fresh and oxidized fish should be susceptible to chemical modification by bisulfite which adds to aldehydes and many ketones to form nonvolatile bisulfite-addition products (Morrison and Boyd, 1978).

Cellulose

Cellulose is a polymer of glucose connected by β1-4 glucosidic bonds. The polymers are laid down naturally in crystalline areas which vary in size and are held in lateral associations by hydrogen bonds. Between crystalline areas are non-crystalline (amorphous) areas which can be hydrolyzable by acids and enzymes (Glicksman, 1969). Some researchers (Gilkes et al., 1992; Geluk et al., 1992; Jach and Sugier, 1983; Laidsaar et al., 1981) have reported that cellulose could be used to adsorb enzymes such as cellulase, lipase, and glucoamylase and to immobilize the enzymes as the catalyst for several bioconversions.

Saito et al. (1992) used cellulose to adsorb carthamine (a red colorant) for its isolation and partial purification. Cellulose was used as a adsorbent for quantitative headspace analysis of volatile organic

compounds (α-pinene, pyridine, ethyl acetate, and methyl ethyl ketone) to investigate the nature of flavor interactions with food ingredients (Saleeb and Pickup, 1978). Gupta et al. (1979) examined the capacity of cellulose to adsorb diacetyl from beer.

Lactose

Lactose has served as an extender for spices and volatile aromas (Reger, 1958). At one time it was used in the manufacture of instant coffee to adsorb volatiles during roasting and drying. When this lactose was incorporated into the coffee powder the flavor was improved (Nickerson, 1979). Maier (1969, 1970, 1972) studied binding of various vapors onto lactose, pectin, and ovalbumin. Knapp (1973) studied a hydrocolloid-stabilized lactose-flavor system, and Yabumoto et al. (1975) have investigated the adsorption and rate of desorption of banana volatiles on lactose. The strengths of adsorption of numerous small organic molecules on anhydrous α- lactose have been measured by McMullin et al. (1975). Adsorptive behaviors of several low molecular weight compounds (commonly associated with food flavor) onto stable anhydrous α-lactose were studied by Marvin et al. (1979) and they found that aromatic hydrocarbons (toluene and ethylbenzene) were adsorbed the least, while alcohols (1-pentanol and 1-octanol) were adsorbed in the greatest amounts; esters and ketones showed intermediate adsorption. Nickerson (1979) found that anhydrous lactose could adsorb a large variety of volatile compounds. Traps containing lactose as an adsorbent were found to be a very simple method of collecting volatile compounds from gas chromatographic effluents for organoleptic evaluation

(Gramshaw, 1976). McMullin et al. (1975) reported that a wide variety of organic flavor compounds including esters, aldehydes, ketones, alcohols and hydrocarbons, were adsorbed onto stable anhydrous α-lactose. Nickerson and Dolby (1971) examined and compared the adsorption of diacetyl on various forms of lactose and other carbohydrates and concluded that for lactose the greatest adsorption occured on anhydrous α lactose.

Pectin

Pectin is composed primarily of D-galacturonic acid, although it can have other carbohydrate moieties linked to it. Partial methylation of the carboxyl groups on the galacturonic acids imparts important properties to pectic substances. Pectin was found to be capable of adsorbing organic molecules, including bile acids, cholesterol, and toxic compounds (Schneeman, 1986).

Guichard et al. (1991) studied the influence of the amount of pectin added on strawberry jam, and the degree of esterification as well as molecular weight of that pectin on sensory characteristics, and on amounts of volatile compounds in the package headspace.

Sensory Evaluation

Sensory analysis is the scientific discipline that evokes, measures, analyzes, and interprets reactions to product characteristics through the senses of sight, smell, taste, touch, and hearing (Stone and Sidel, 1985). The role of sensory evaluation is to provide valid and reliable information to research & development, production and marketing in order for management to make sound

business decisions about the perceived sensory properties of products. The ultimate goal of any sensory program should be to find the most cost-effective and efficient method with which to obtain the most sensory information (Meilgaard et al., 1987).

The sensory attributes of a food item are: appearance, odor/aroma/fragrance, consistency and texture, and flavor (aromatics, chemical feeling, taste) (Meilgaard et al., 1987).

In order to measure true product difference three major variables must be controlled. The first, test controls: the test room environment, the use of booths or a round table, the lighting, the room air, the preparation area, the entry and the exit areas. The second, product controls: the equipment used, the way samples are screened, prepared, numbered, coded and served. The third, panel controls: the procedure to be used by a panelist evaluating the sample in questions (Meilgaard et al, 1987).

Factors influencing sensory verdicts are physiological factors (adaptation, enhancement or suppression), psychological factors (expectation error, error of habitation, stimulus error, logical error, halo effect, order of presentation of samples, mutual suggestion, lack of motivation, and capriciousness vs. timidity), and poor physical condition (Meilgaard et al, 1987).

The first quality judgement made by a consumer about a fish product at the point of sale is based on its appearance. The next judgement is about the product's texture. This is followed by an odor judgement, and finally by taste to judge flavor (Stone and Sidel, 1985).

Connell and Howgate (1971) evaluated the flavor/texture impact on the acceptance of cod and haddock fillets over a wide range of freshness and concluded that flavor was a more important criterion of quality than texture. Similarly, Hamilton and Bennett (1983, 1984), in studies using different species of white fish, concluded that flavor was the most significant positive determinant of acceptability. Laslett and Bremner (1979) conducted a series of storage trials with minced flesh from Australian fish species and concluded that off-flavor, off-aroma and flavor were just as important as texture variables in determining the acceptability of frozen fish products. Kelly (1969) reported that the acceptability of frozen cod was affected more by the development of cold storage flavors than by textural changes.

Human perceptions of food are complex and may be influenced significantly not only by the sensory characteristics of the food material, but also by the personal experience of the observer such as his or her physiological/nutritional state, psychological state, ethnic origin, educational level, economic level, religious convictions, social/cultural pressures, and so on. Apparently "normal" individuals may differ substantially in their internal anatomy, and in various biochemical and physiological functions (Williams, 1971). Such differences can have significant impact on the individual's food behavioral patterns. How the brain integrates all of these influences, sensory and others, determines a person's response (acceptance/rejection; pleasantness/ unpleasantness) to the food.

Acceptability of foods, the degree of liking and disliking, is usually estimated using a scalar method, the most common one being

the nine-point structured hedonic scale(Peryam 1952). Validity of data generated using this method can be influenced by factors (in addition to those already mentioned) such as unequal size category intervals in the scale, the tendency of panelists to avoid extreme values on the scale and to score close to the midpoint, the presence of restrictive end points on the scale, and so on. Nevertheless, this method, or variations of it, is probably the most widely used technique for estimating hedonic quality of foods.

A method commonly used for consumer preference testing is the two sample or paired comparison test. This is a very simple test method that is easily understood by the participant. In this test, a participant is asked only to indicate which one of the two samples he or she prefers. Data generated using this procedure, however, yield no information about the degree or intensity of the preference. If information on the magnitude of acceptability, preference, and so on is desired, some kind of scaling technique must be utilized.

Analytical testing to determine differences or similarities in food products, to identify quality attributes, and to estimate relative intensity of sensory attributes is usually accomplished in a laboratory setting, under controlled conditions, with a trained panel of subjects. There are numerous reviews in the literature that describe in depth aspects of physical facilities, sample handling, selection and training of panelists, data analysis, and so on(Stahl and Einstein, 1973; Larmond, 1977; Amerine et al., 1965). Difference tests are designed to identify samples that differ in terms of a sensory characteristic. The attribute must be defined in the single sample and in two samples (paired comparison) test methods, but

need not be defined with the duo-trio (matching one of two coded samples with a labeled reference sample) and triangle (two samples identical, one different) methods. These are commonly used "difference" test methods. The information they yield is limited and if intercomparison of several samples is needed, an extensive amount of testing may be required, especially if tests are replicated to check on reliability of panel performance. If sensory analytical tests are properly conducted, with appropriate control of operational variables, they can yield reliable and reproducible data.

MATERIALS AND METHODS

Materials

Fish

Lake trout (*Salvelinus namaycush*) is classified as high oil-low protein fish (Stansby and Olcott, 1963). Oil content in lake trout is about 15%. Lipid oxidation in lake trout is a serious problem to quality of fish. Four (from the same batch) whole , uncooked, gutted and gilled frozen lake trout were purchased from Superior Seafoods Co., Grand Rapids, Michigan. At the time of purchase, the fish were frozen and wrapped in polyethylene film and then packed in a corrugated box. The fish were iced in a cooler and transferred back to E. Lansing. It was about an hour and half in the cooler. The fish with the wrap were then removed from the cooler and stored in the freezer at the Food Lab , Department of Food Science and Human Nutrition. One was prepared for identification of volatile aroma compounds, three fish were used for evaluation of the selected adsorbents, and the other one was cut into small cubes for sensory evaluation.

Adsorbents

1. Silica gel, ACS reagent*, blue-indicating, Aldrich Chemical Company, Inc. (Milwaukee, Wisconsin).

2. Activated Carbon, Darco® 20-40 mesh, granular, general-use carbon from coal raw materials, Aldrich Chemical Company, Inc. (Milwaukee, Wisconsin).
3. Sodium bisulfite, ACS reagent, granular, analytical reagent, Mallinckrodt, Inc.(Paris, Kentucky).
4. Cellulose, powder ~20micron average particle size, Aldrich Chemical Company, Inc.(Milwaukee, Wisconsin).
5. Pectin, analytical grade, Nutritional Biochemicals Corporation. (Cleveland, Ohio).
6. Anhydrous α-lactose, analytical grade, Aldrich Chemical Company, Inc.(Milwaukee, Wisconsin).

*ACS reagents meet specifications and test requirements set by the American Chemical Society.

Volatile Standards

1. Butanal (Butyraldehyde), 99% purity, FW72.11, m.p.-96°C, d 0.800, b.p.75°C.
2. Cycloheptatriene, 90% purity, FW92.14, d 0.888, b.p.116-117°C.
3. Hexanal (Caproaldehyde, Hexaldehyde), 98% purity, FW100.16, d 0.834, b.p.131°C.
4. Pentanal (Valeraldehyde), 99% purity, FW86.13, d 0.810, b.p.103°C.

All volatile standards were purchased from Aldrich Chemical Company (Milwaukee, Wisconsin).

Packaging Material for Adsorbents

The adsorbents were packed in Tyvek pouches (1.0in.x1.0in.) which were sealed using the impulse heat sealer. The packaging material, Tyvek, is made by du Pont de Nemours & Co. Tyvek is a low oxygen barrier material.

Pierce Mininert®[1] Valve (as shown in Figure 1.) (Pierce, Rockford, I.L.)

The body of the valve was made of Teflon®[2]. The pressure seal was made at the mouth of the cell using a system which consisted of two rubber rings encased inside the base of the valve. Pressure was exerted using a threaded ring. The ring was turned down on the rubber rings which caused the base of the valve to bulge, making the seal. It provided a pressure tight seal to at least 1.5 atmosphere. The valve mechanism was created using a sliding cylinder which passed through the axis of the valve at right angles. When the valve was opened, a needle could be inserted through the valve's axis into the diffusion/adsorption cell. When the valve was closed, the hole in the sliding cylinder was no longer in line with the valve axis and a needle could not be pushed though it. To prevent gas from escaping around the needle when it was inserted, a small silicone septum (Pierce, Rockford,I.L.) was located above the sliding cylinder.

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[1]Registered Trademark, Pierce, Rockford, I.L.

[2]Registered Trademark, I.E. du Pont de Nemours & Co.

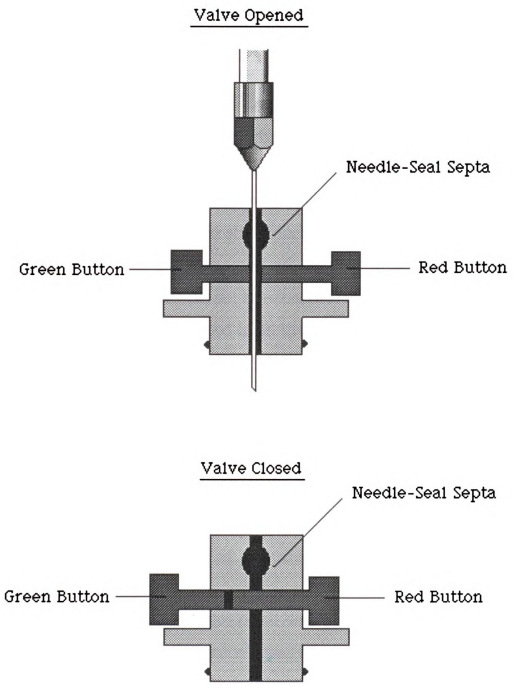

Figure 1. Pierce Mininert valves: Operation is simple- just push the green button to open, insert syringe needle and take sample, withdraw needle, then push red button to colse. To change needle-seal septa, simply push the old septum out with 1/8" diameter rod and push a new cylindrical septum in. This is done with the valve colsed to prevent exposure of contents.

Methodology

This project was designed to evaluate the capacity of six chemicals, including silica gel, activated carbon, sodium bisulfite, cellulose, pectin and lactose to adsorb off-flavors associated with oxidized frozen fish. Three experiments were conducted to achieve this. The first experiment was designed to identify the volatile compounds of oxidized frozen fish. The next experiment was to distinguish the ability of the six adsorbents using a model system which incorporated the suspected off-flavor compounds identified from experiment I with the six adsorbents. In experiment III, the selected adsorbent was sealed individually in Tyvek pouches and stored with a sample of oxidized frozen fish in a glass jar. By comparing the concentration of the volatile compounds in the headspace, the most effective adsorbent among the six chemicals was determined. Sensory evaluation was done to demonstrate the effect of the selected adsorbent on the level of adsorption of off-odors.

Experiment I. Identification of Volatile Aroma Compounds from Oxidized Frozen Fish

Extract from frozen lake trout was prepared by immersing a whole (3 lbs), thawed, uncooked lake trout in 500 ml of a saturated sodium sulfate solution, followed by agitating to recover most of the slime layer in the extract. Several extracts were pooled to obtain a large sample of the drip/slime mixture, and 100 ml subsamples were taken for analysis using the bubble-purge configuration apparatus (250 ml flask) as shown in Figure 2 (Olafsdottir, 1985). Headspace volatiles from the extract were collected and concentrated by

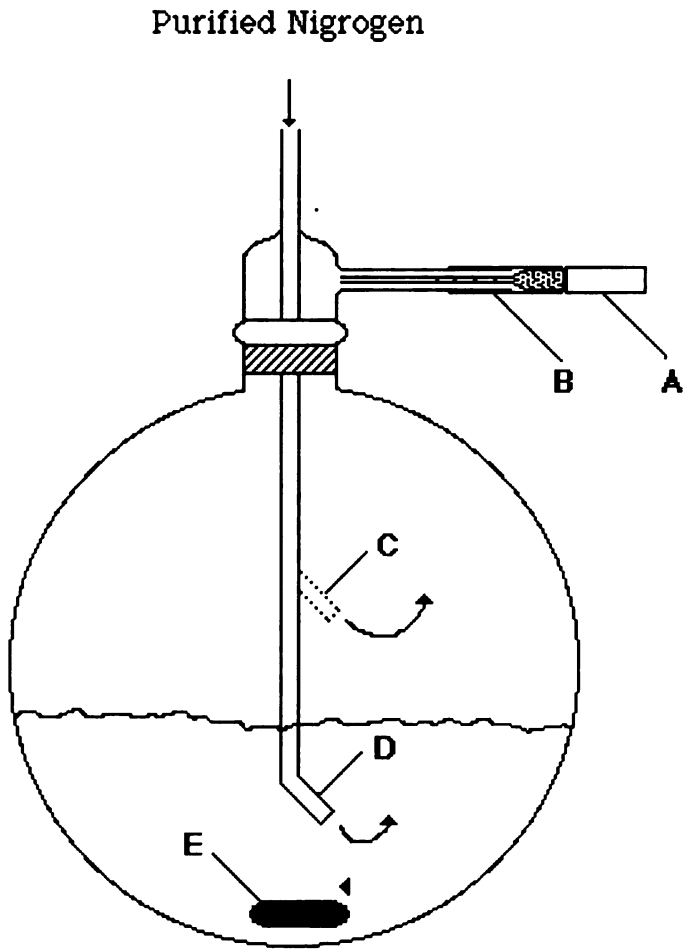

Figure 2. Glass apparatus for dynamic gas-purging of headspace aroma volatiles onto Tenax-GC:
(A) Tenax-GC collection tube;
(B) Heat-shrinkable Teflon tubing;
(C) Configuration of purge-tube for swept-surface sampling;
(D) Configuration of purge-tube for bubble sampling; and
(E) Magnetic stirring bar.

purging the sample with nitrogen (100 ml /min for 3 hours) at 21°C onto Tenax GC as described by Steinke (1978).Capillary column GC in conjunction with mass spectrometric analyses of volatiles in ethyl ether extracts from Tenax GC traps was performed as reported by Josephson et al., (1983a). Identification of compounds was based on coincidence of mass spectral patterns of standards.

Mass Selective Detector (MSD), Hewlett-Packard 5970 MSD Mass Spectroscopy, was used to identify the off-flavor compounds. The conditions of MSD were as follows:

Detector: Picogram range

Sample compatibility: Mass range 10 - 800 amu (He/H_2 excluded) Volatility stability: Max interface To 320°C

Sample introduction: Capillary direct from Hewlett Packard 5890 GC (Option: open-split interface)

Quadrapole mass filter

Solvent delay: 3.00 min.

Resulting voltage: 2000 e.v.

Ion source: 70 e.V.

Run parameter: Start time 3.00 min.

Low mass 40 amu

High mass 400 amu

Scan threshold 1000

a/d sample (2^n): 2

Scan/sec 1.19

Temperature zone: Initial temperature 40°C

Initial time 0 min.

Rate 5°C/min

Final temperature 60°C

Final time 0 min.

Rate A 10°C/min

Final temperature A 280°C

Final time A 30 min.

Injection port temperature: 250°C

Transfer line temperature: 250°C

Hewlett-Packard 5890 GC: Carbowax 20M (Supelco Inc., Bellefonte, PA)

Fused silica capillary column

Column dimensions: 60mX0.31mm

Head pressure: 50 psi

Split vent flow: 1.00 ml/min

Septum purge flow: 7 ml/min

Experiment II. Distinguishing the Adsorbing Capability of Six Perspective Adsorbents

The adsorbing capacity of six perspective adsorbents: silica gel, activated carbon, sodium bisulfite, pectin, lactose, and cellulose, were investigated using the techniques developed by Oosting et al., (1984).

The volatile standards were butanal, cycloheptatriene, pentanal, and hexanal which were the major off-flavor volatiles identified from Experiment I. Essentially, 5 µl of the liquid volatile standards were injected into a 125ml serum bottle fitted with a

Pierce Mininert Valve^{R} as shown in Figure 3. The adsorbent (2.0gm), enclosed in a Tyvek (by DuPont) pouch (1.0in.X1.0in.), was suspended above the volatile standards. Samples of volatile standards in the headspace were withdrawn through the Pierce Mininert Valve^{R}, and the uptake of the vaporized sample by the adsorbents was monitored over several time intervals (24, 48, and 72 hours) at 21^{o}C. A 10 µl Hamilton liquid sampling syringe was used to sample the volatile standards, and a 250µl Hamilton gas tight sampling syringe was used to sample the headspace.

A 125ml serum bottle was used as the diffusion/adsorption cell. 5 µl of volatile standards were withdrawn and injected into the diffusion/adsorption cell. A Pierce Mininert Valve^{R} was fitted to the cap of the cell. To prevent gas from escaping around the needle when it was inserted, a small silicone septum was located above the sliding cylinder at the point where the needle was inserted.

The headspace of the diffusion/adsorption cell was sampled using a 250µl Hamilton gas tight syringe. The syringe was cleaned by aspiration of acetone through the needle and heated at 70^{o}C for 30 minutes. Prior to sampling the vapor, the syringe was conditioned to room temperature. The syringe needle was plunged through the Pierce Mininert Valve® into the bottle. About 250µl of the headspace was drawn into the syringe and expelled back into the bottle. Then 25µl of the vapor was withdrawn and quickly injected onto the gas chromatographic column.

Analysis of headspace concentrations was conducted using a gas chromatographic procedure. A Hewlett-Packard Model 5890 w/split vent, equipped with dual flame ionization detectors and

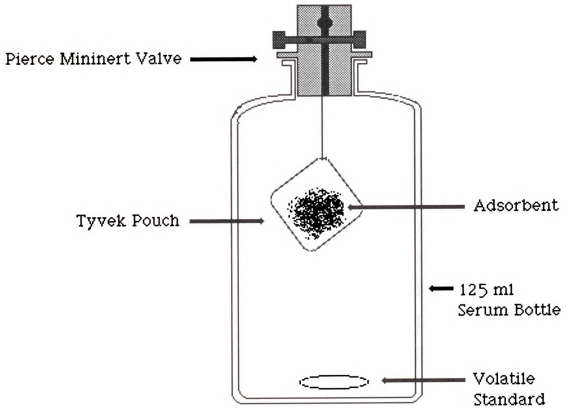

Figure 3. Cross Sectional View of Diffusion/Adsorption Cell

interfaced to a Hewlett-Packard Model 3392A integrator was employed. The gas chromatographic conditions were as follows:

Column: DB-225 (J&W Scientific, Folsom, California)

Fused silica capillary column

Column dimensions: 30mX0.249mm

Temperature limits: 40°C to 220°C

Column flow rate=$0.785xD^2L/t_r$=$0.785x(0.25)^2x30/1.87$

=0.7871

Split Ratio=(4.26+0.7871)/0.7871=6.41

Temperature: Injection temperature 200°C

Detector temperature 250°C

Oven temperature 150°C

Temperature programme:Initial temperature 40°C

Initial time 5 min.

Rate 2°C/min

Final temperature 48°C

Final time 0 min.

Rate A 10°C/min

Final temperature A

200°C

Final Time A 40 min.

Integrator Model 3392A

Zero:0,-0.9

Attenuation: 2

Chart speed: 0.5cm/min

Peak width: 0.04

Threshold: 0

Experiment III. Evaluation of the Selected Adsorbents with Frozen Fish System at Low Temperature (5°C)

Three whole, uncooked, frozen lake trout were thawed. Each of the fish was ground into a large sample and 40 grams of the ground fish sample were placed in a 450 ml glass jar with a screw cap which had a sampling port on the top as shown in Figure 4 . Two grams adsorbent, silica gel or activated carbon, were packed in a Tyvek pouch (1.0in.x1.0in.) and suspended above the fish sample for three days in the walk-in cooler (5°C). A 250 µl gas tight syringe was used to sample the headspace. 200 µl of the vapor in the headspace were withdrawn and quickly injected onto the GC column.

Analysis of headspace concentrations was carried out using a gas chromatographic procedure. A Hewlett-Packard Model 5890A gas chromatograph, equipped with dual flame ionization detectors and interfaced to a Hewlett-Packard Model 3392A integrator was employed. The gas chromatographic conditions were as follows:

Column: Supelcowax10 (Supelco Inc., Bellefonte, PA)
Fused silica capillary
Polar bonded stationary phase
Column dimensions 60mx0.25mmI.D.

Carrier gas: Helium at 27ml/min

Temperature: Injection temperature 200°C
Detector temperature 250°C
Oven temperature 150°C
Temperature programme:Initial temperature 40°C
Initial time 1 min.

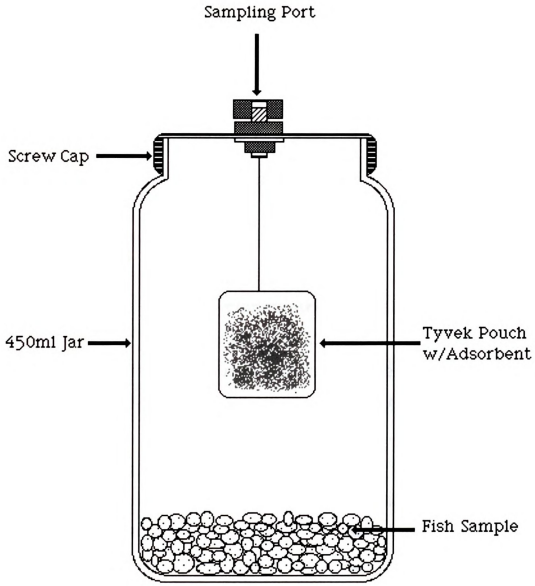

Figure 4. Cross Sectional View of Diffusion/Adsorption Jar

Rate 5°C/min

Final temperature 220°C

Final time 10 min.

Integrator Model 3392A

Zero: 0, -1.1

Attenuation: 2

Chart speed: 0.5cm/min

Peak width: 0.04

Threshold: 0

Retention time(R_T) of Butanal: 2.08min

Pentanal: 3.09min

Cycloheptatriene: 4.55min

Hexanal: 5.32min

The standard calibration curves for butanal, pentanal, cycloheptatriene, and hexanal are shown in Appendix B.

Sensory Evaluation

A simple paired comparison test was used for sensory evaluation. Frozen lake trout was cut into small cubes. 10 grams of these cubes were placed in a 450ml glass jar which was previously described in Section III. A Tyvek pouch (1.0in.x1.0in.) packed with glass beads was suspended above the fish cubes in the glass jar as the control.

The selected adsorbent, activated carbon, was packed in a Tyvek pouch(1.0in.x1.0in.) and hung above the 10 grams of fish cubes in the 450ml glass jar as the treatment.

Three pairs of samples (one control and one treatment as a pair) were prepared for each panelist. Thus, totally 36 jars of samples were prepared for six panelist. Also three pairs of 3-digit random numbers were chosen to code the jars. The panelists had not been informed the representation of the codes.

All samples were stored at 5°C for 3 days and then transferred to the Food Lab in the School of Packaging where the testing sessions were held. The Food Lab was equipped with standard indoor fluorescent lighting and the temperature was at 21°C.

Six panelists included faculty and graduate students from the School of Packaging and Department of Food Science. All were semi-trained in off-odor intensity and well experienced in sensory evaluation.

During the test sessions (three sessions for each panelist), two 3-digit random number coded jars with the samples (one control and on treatment) as well as the consent form and questionnaire (shown in Appendix E) were presented to each panelist. They were asked to follow the procedures listed in the questionnaire to sniff the samples and identify the sample which had the stronger off-odor and to indicate it on the questionnaire. 36 samples (three replications) had been sniffed, a total of 18 judgments were made.

RESULTS AND DISCUSSION

Major Off-flavor Volatiles of Oxidized Frozen Lake Trout

Preliminary tests were conducted to identify the off-flavor volatiles associated with oxidized frozen lake trout. The frozen lake trout used in this study exhibited distinct oxidized aromas. Capillary GC in conjunction with mass spectrometry was used to identify the volatiles in ethyl ether extracts from Tenax GC at 21°C and are shown in Table 1. The mass spectra are shown in Appendix A. Butanal, cycloheptatriene, pentanal, and hexanal were found to be the major off-flavor volatiles. Volatile aldehydes have been believed to contribute strongly to the characteristic aroma of oxidized fish lipids (Lea, 1953; Yu et al., 1961; Aiken and Connell, 1979; Ikeda, 1980). Hexanal was found to be the most abundant volatile compound in oxidized frozen whitefish (*Coregonus clupeaformis*) (Josephson et al.,1984). Hexanal has historically been recognized as a principal compound formed in oxidizing lipid systems (Forss et al., 1960; Yu et al., 1961; Badings, 1970; Matthews, 1971; Nobel and Nawar, 1971; Chan et al., 1976; Warner et al., 1978; Selke et al., 1980; Schieverle and Grosch, 1981; Frankel et al., 1982). Yu et al. (1960) found that pentanal and butanal were major volatile compounds in autoxidized salmon oil. Josephson et al. (1984) reported that pentanal was a volatile aroma compound from oxidized frozen whitefish.

RESULTS AND DISCUSSION

Major Off-flavor Volatiles of Oxidized Frozen Lake Trout

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Table 1. Major Volatile Compounds Identified in Frozen Lake Trout

Compounds	Confirmation
Butanal	G.C.[1] M.S.[2]
Cycloheptatriene	G.C. M.S.
Pentanal	G.C. M.S.
Hexanal	G.C. M.S.

1. G.C. confirmation was based on the retention time comparison to the volatile standards.
2. M.S. confirmation was based on comparison to the volatile standards mass spectrum.

Adsorption of Volatile Standards by the Six Adsorbents

Some investigators have found that silica gel was effective in adsorbing volatile compounds (Davis et al., 1952; Ruthven, 1984; Peers and Coxon, 1986; Ackman and Ratnayake, 1989b; Shantha and Ackman, 1991). It has been reported that activated carbon has great adsorption capacity for volatile organics (Tausig and Drake, 1959; Gernon et al., 1961; Matsukura et al., 1984; Ruthven, 1984; Sakaki et al., 1984; Welsh and Zall, 1984; Seo and Morr, 1985; Toro-Vazquez et al., 1991). Yasuhara and Shibamoto(1989) used sodium bisulfite to adsorb hexanal, heptanal, and pentanal. Some investigators have reported that lactose has the ability to adsorb aromas (Nickerson and Dolby, 1971; McMullin et al., 1975; Marvin et al., 1979; Nickerson, 1979; Ehler et al., 1979). Few studies have focused on cellulose and pectin as a adsorbent to adsorb off-flavor compounds, but they are food ingredients. Silica gel, activated carbon, and sodium bisulfite do not have as wide approval to be incorporated into food. Therefore, three food grade carbohydrates (cellulose, lactose, and pectin), and three non-food grade chemicals (silica gel, activated carbon, and sodium bisulfite) were chosen as adsorbents in this preliminary report to determine the potential use of these six compounds for adsorbing off-flavor volatile aromas in oxidized frozen lake trout.

Analysis of the headspace in the serum bottle was done every 24 hours from time zero to 72 hours using GC. Area responses from each GC analysis are shown in Appendix C (Table C-9).

The concentration of volatile standard in the headspace is directly proportional to the area response. High area response means

high concentration, and thus adsorption of the adsorbent was less effective. For each volatile standard, the highest recorded area response is proportional to 100% relative concentration of the volatile standard in the headspace. The data in Table C-9 was converted to the relative % concentrations as shown in Table 2.

Effectiveness of Six Adsorbents on Butanal

The effectiveness of six adsorbents on butanal was evaluated every 24 hours up to 72 hours after storage at 21°C and the results were shown in Table 2.

After 24 hours storage at 21°C with activated carbon as the adsorbent, the relative % concentration of butanal in the vial headspace was 0.39% which means that activated carbon adsorbed most of the butanal in the vial headspace. Silica gel also adsored a high level of butanal. As the results show, 1.74% butanal was detected in the vial headspace. Sodium bisulfite did not demonstrate as high adsorption capacity as did activated carbon and silica gel, but it was considered a good adsorbent for butanal. Almost 70% of the butanal was adsorbed by sodium bisulfite. Cellulose, lactose, and pectin showed poor adsorption of butanal. On the second day (48 hours storage), activated carbon still had the highest level of adsorption and the relative % concentration of butanal was reduced from 0.39% to 0.19%. Silica gel also adsorbed butanal very well. The relative % concentration of butanal was reduced from 1.74% to 0.66%, while sodium bisulfite, cellulose, lactose, and pectin demonstrated small reductions in the relative % concentration of butanal after 48 hours storage.

Table 2. Relative % Concentration of Volatile Standards in the Vial Headspace after 24, 48, and 72 Hours Storage at 21°C

Adsorbent	Time (hrs)	Butanal	Cyclohepta-triene	Pentanal	Hexanal
Silica gel	24	1.74	0.23	ND*	58.12
	48	0.66	0.16	ND	23.16
	72	0.15	ND	ND	8.66
Activated carbon	24	0.39	ND	ND	47.64
	48	0.19	ND	ND	12.11
	72	0.10	ND	ND	8.26
Sodium Bisulfite	24	29.64	98.56	94.16	72.35
	48	21.17	81.06	78.94	36.61
	72	10.88	80.22	72.36	6.95
Cellulose	24	77.16	89.57	83.08	63.03
	48	64.24	81.30	71.57	35.78
	72	53.78	73.20	58.21	24.04
Lactose	24	91.01	97.82	93.55	62.02
	48	81.20	91.91	80.70	45.19
	72	71.91	79.82	60.93	30.58
Pectin	24	97.29	95.12	95.95	86.95
	48	90.84	83.81	80.16	62.53
	72	76.46	76.68	70.41	43.12

*ND: Not Detectable

Among all six adsorbents, activated carbon was the most effective on day 3 (72 hours storage), followed by silica gel, while sodium bisulfite, cellulose, lactose, and pectin indicated no substantial adsorption of butanal.

Activated carbon and silica gel were the most effective adsorption of butanal (Figure 5), followed by sodium bisulfite, cellulose, lactose, and pectin.

Effectiveness of Six Adsorbents on Cycloheptatriene

Relative % concentration of cycloheptatriene in the vial headspace are shown in Table 2. No significant adsorption occurred with sodium bisulfite, cellulose, lactose, and pectin, while activated carbon and silica gel demonstrated distinctive adsorption of cycloheptatriene during 72 hours storage at 21°C.

As shown in Figure 6, there were significantly different adsorption profiles between the two groups of adsorbents. Activated carbon and silica gel had the most significant adsorbing effect on cycloheptatriene. Sodium bisulfite, cellulose, lactose, and pectin were much less effective than activated carbon and silica gel.

Effectiveness of Six Adsorbents on Pentanal

As shown in Table 2, pentanal was not detectable in the presence of silica gel or activated carbon during three days storage at 21°C. Silica gel and activated carbon were very effective in adsorbing pentanal. Sodium bisulfite, cellulose, lactose, and pectin exhibited poor adsorption of pentanal after 24 hours storage at 21°C. After 72 hours, there was approximately 30% pentanal in the vial

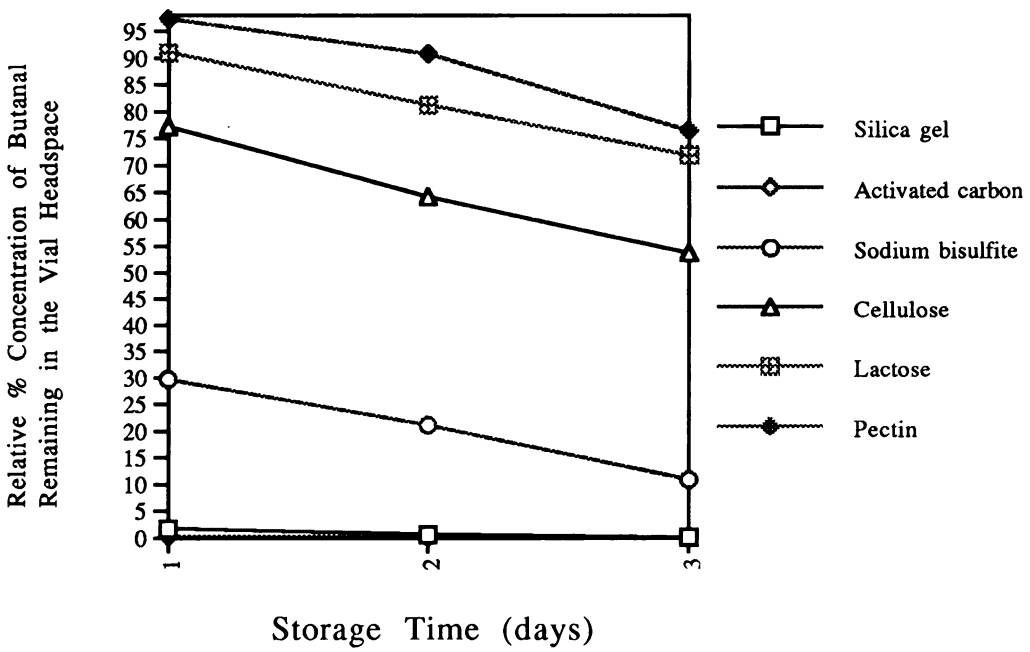


Figure 5. Adsorption by Adsorbents of Butanal during Storage at 21°C

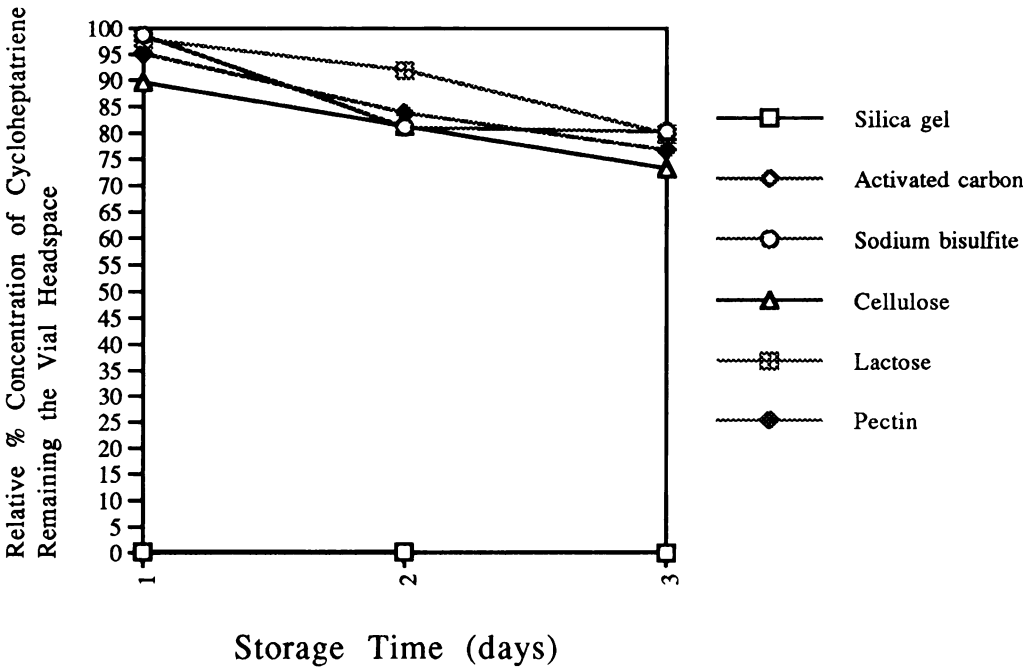


Figure 6. Adsorption by Adsorbents of Cycloheptatriene during Storage at 21°C

headspace containing sodium bisulfite, and pectin had about the same level as sodium bisulfite. Cellulose exhibited more than 40% adsorption of pentanal after 72 hours storage at 21°C. Lactose adsorbed approximately 40% pentanal in the vial headspace by day 3.

Silica gel and activated carbon (Figure 7) exhibited very high adsorption of pentanal, while sodium bisulfite, cellulose, lactose, and pectin exhibited some adsorption of pentanal. Among these four adsorbents, cellulose was better than the other three. During the first two days, there was no significant difference among sodium bisulfite, lactose, and pectin. On day 3, lactose was more effective than sodium bisulfite and pectin.

Effectiveness of Six Adsorbents on Hexanal

Generally, the relative % concentration of hexanal in the vial headspace results (Table 2) were different from the previous volatile standards. After one day storage (21°C), almost 50% of the hexanal was adsorbed by activated carbon, while silica gel, cellulose, and lactose adsorbed about 40% hexanal. Sodium bisulfite adsorbed approximately 30% hexanal after 24 hours storage at 21°C. Pectin showed poor adsorption of hexanal, with only 13% hexanal being adsorbed.

The adsorption by silica gel, activated carbon, and sodium bisulfite of hexanal greatly increased, while cellulose, lactose, and pectin demonstrated moderate changes after 48 hours storage at 21°C (Table 2). With silica gel as the adsorbent, the relative % concentration of hexanal was reduced from 58% (24 hours storage) to

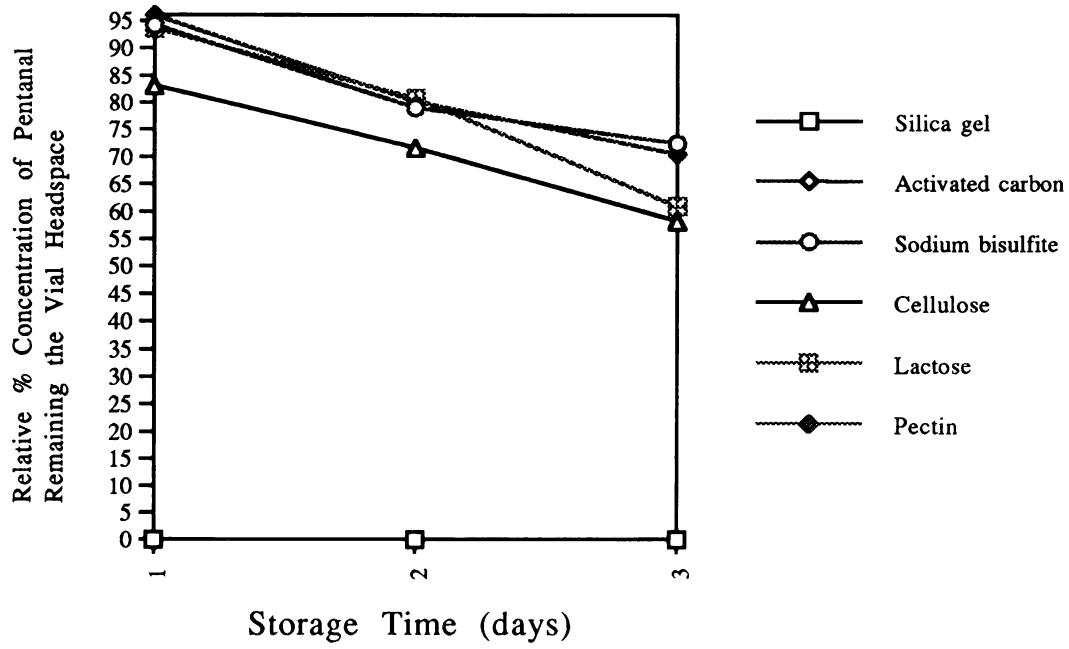


Figure 7. Adsorption by Adsorbents of Pentanal during Storage at 21°C

23% (48 hours storage). Activated carbon lowered the level of hexanal in the vial headspace from 48% to 12%. The relative % concentration of hexanal in the vial headspace was reduced from 72% (24 hours storage) to 37% (48 hours storage) in the presence of sodium bisulfite as the adsorbent. Cellulose showed an increase in adsorption of hexanal from 63% (24 hours storage) to 36% (48 hours storage). With lactose as the adsorbent, 45% hexanal was measured in the vial headspace after 48 hours storage at 21°C. Pectin exhibited low adsorption capacity of hexanal with a 63% relative concentration of hexanal remained in the vial headspace after 48 hours storage at 21°C (Table 2).

After 72 hours storage at 21°C, sodium bisulfite, activated carbon, and silica gel had the most effective adsorption of hexanal (7% for sodium bisulfite, 8% for activated carbon, and 9% for silica gel) (Table 2). Cellulose exhibited good adsorption of hexanal on day 3 (72 hours storage), 24% relative concentration of hexanal (Table 2) remaining in the vial headspace. Approximately 70% of the hexanal was adsorbed by lactose after 72 hours storage at 21°C, and about 60% hexanal was adsorbed by pectin under the same storage conditions.

As shown in Figure 8, all adsorbents adsorbed hexanal. On day 1, activated carbon had the greatest adsorption of hexanal followed by silica gel, cellulose (lactose), sodium bisulfite, and pectin in that order. On day 2, activated carbon still had the highest adsorption of hexanal. On day 3, sodium bisulfte, activated carbon, and silica gel had the most adsorption of hexanal.

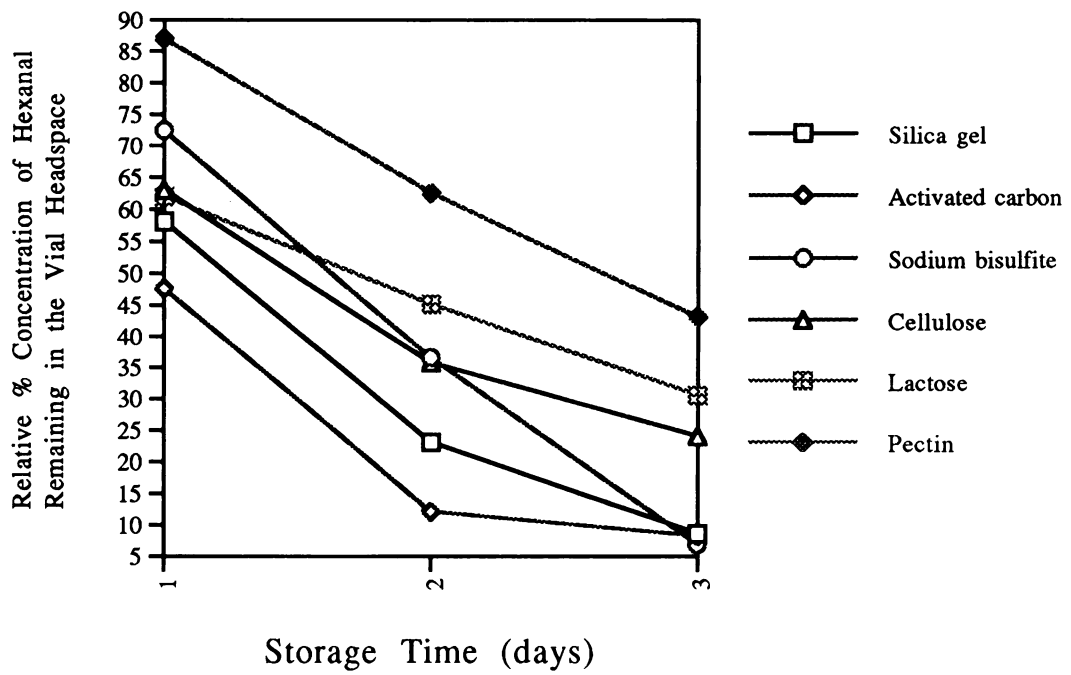


Figure 8. Adsorption by Adsorbents of Hexanal during Storage at 21°C

Overall, silica gel, activated carbon, and sodium bisulfite exhibited outstanding adsorption of butanal (Figure 9). For cycloheptatriene and pentanal, silica gel and activated carbon had the greatest adsorption capacity of the adsorbents (Figure 10, 11). On day 1, the effect of the adsorbents on hexanal (Figure 12) was about the same. On day 2, activated carbon demonstrated the greatest adsorption, while sodium bisulfite had more adsorption on day 3.

As shown in Figure 13, silica gel adsorbed butanal, cycloheptatriene, and pentanal quite readily. Activated carbon also had excellent adsorption of butanal, cycloheptatriene, and pentanal (Figure 14) , while sodium bisulfite exhibited good adsorption of butanal and hexanal (Figure 15). Cellulose, lactose, and pectin showed almost no or imited adsorption of any of the volatile standards (Figure 16, 17, 18).

Adsorption involves the accumulation of substances at a surface or interface, and occurs in large measure as a result of forces active within surface boundaries. Two types of binding forces are commonly distinguished; i.e., physical and chemical. Physical adsorption results from the action of van der Waals forces, comprised of London dispersion forces and classical electrostatic forces. The second important category of surface interaction is that of chemisorption. As a result of significant affinities, molecular orbital overlap occurs between molecules in the respective phases. Transfer and sharing of electrons take place between adsorbed solute and adsorbent, and the chemisorptive bond can have all the characteristics of a chemical bond. The chemisorptive bond is localized at active centers on the adsorbent and is usually stronger

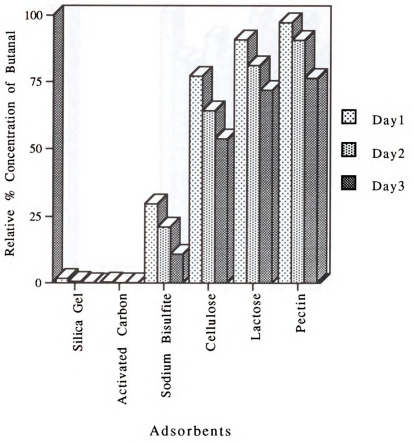


Figure 9. Relative % Concentration of Butanal Remaining in the Vial Headspace

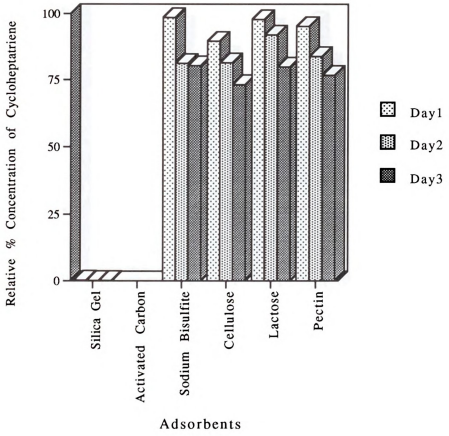


Figure 10. Relative % Concentration of Cycloheptatriene Remaining in the Vial Headspace

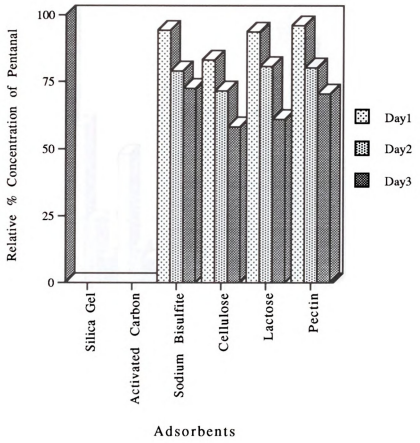


Figure 11. Relative % Concentration of Pentanal Remaining in the Vial Headspace

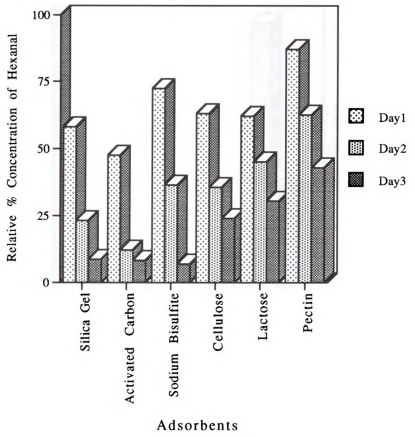


Figure 12. Relative % Concentration of Hexanal Remaining in the Vial Headspace

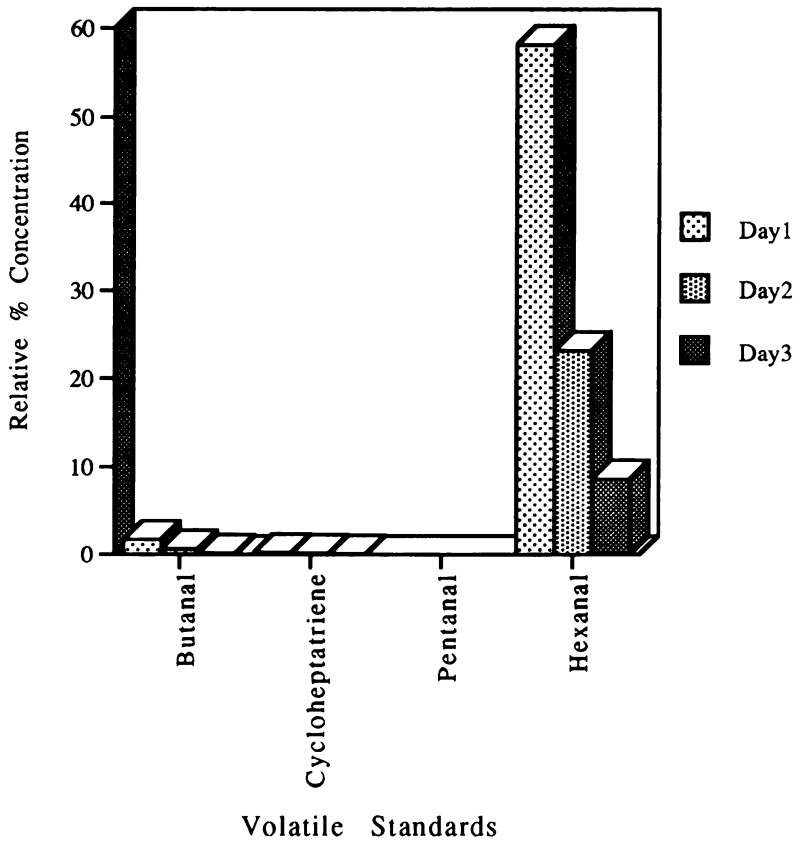


Figure13. Relative % Concentration of Volatile Standards Remaining in the Vial Headspace with Silica Gel as the Adsorbent

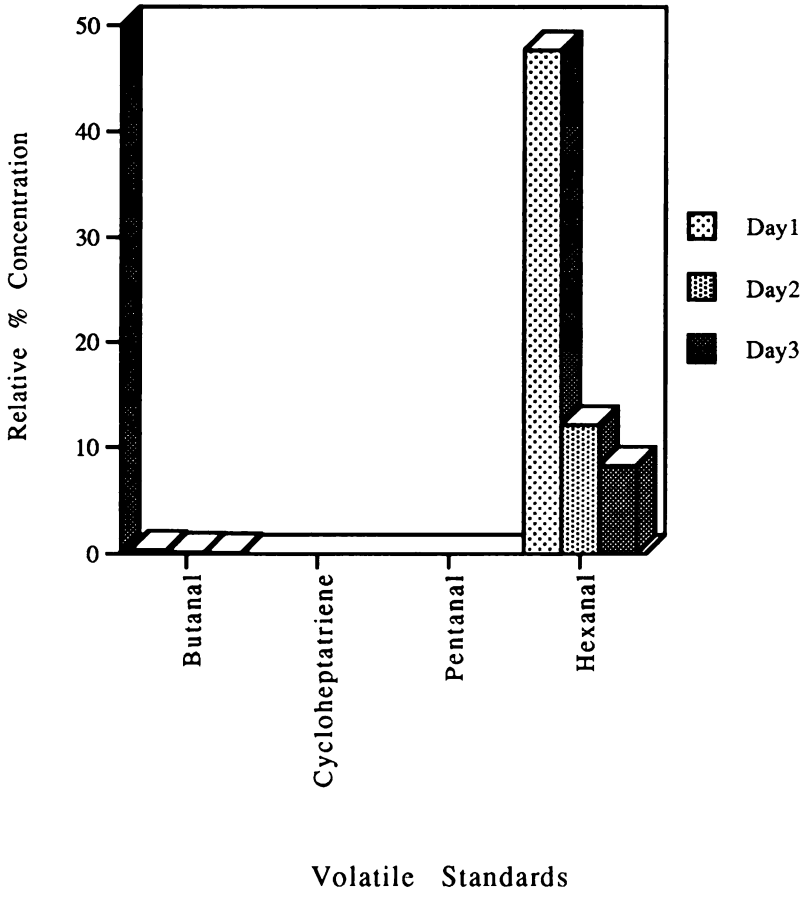


Figure 14. Relative % Concentration of Volatile Standards Remaining in the Vial Headspace with Activated Carbon as the Adsorbent

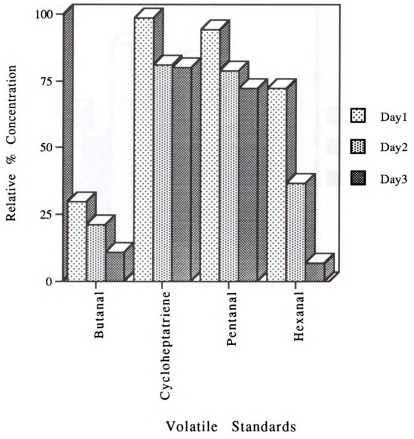


Figure15. Relative % Concentration of Volatile Standards Remaining in the Vial Headspace wtih Sodium Bisulfite as the Adsorbent

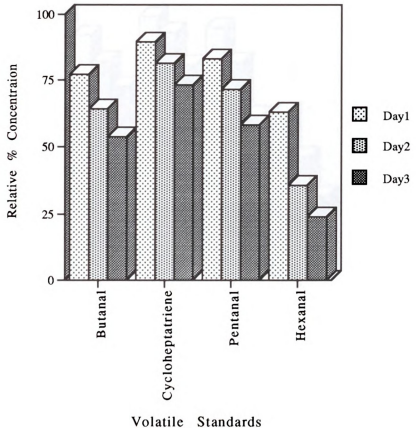


Figure16. Relative % Concentration of Volatile Standards Remaining in the Vial Headspacewith Cellulose as the Adsorbent

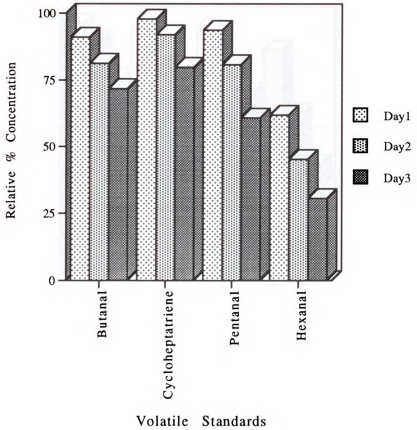


Figure17. Relative % Concentration of Volatile Standards Remaining in the Vial Headspacewith Lactose as the Adsorbent

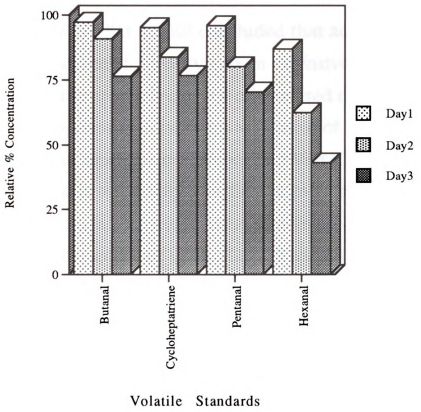


Figure18. Relative % Concentration of Volatile Standards Remaining in the Vial Headspace with Pectin as the Adsorbent

than the physical van der Waals forces. Whereas physical adsorption is usually dominant at low temperatures, chemisorption is favored by higher temperature, since chemical reactions proceed more rapidly at elevated temperatures than at lower temperatures (Weber and Van Vliet, 1980).

Weber and Van Vliet (1980) concluded that activated carbon exhibited a high degree of porosity and an extensive associated surface area. The intraparticle surface of activated carbon is sufficiently heterogeneous to participate in most of the various physical interaction mechanisms (Weber and Van Vliet, 1980). Because adsorption is essentially a surface or interfacial phenomenon, the surface characteristics of activated carbon are of major import (Weber and Van Vliet, 1980). That is the reason why activated carbon had significant adsorbing capacity of volatiles organics.

The presence of hydroxyl groups imparts a degree of polarity to the surface of silica gel so that molecules such as water, alcohols, phenols, amines (which can form hydrogen bonds), and unsaturated hydrocarbons (which can form π- complexes) are adsorbed (Ruthven, 1984). This may explain why silica gel was able to adsorb the volatile standards.

Cellulose, lactose, and pectin also have hydroxyl groups in their chemical structures (McMullin et al., 1975; Schneeman, 1986). These hydroxyl groups are the most likely locations to which a negatively charged adsorbate center would be attracted. It is likely that a hydrogen bond forms (McMullin et al., 1975). However, since the availability of the hydroxyl groups to form hydrogen bonds with the

volatile standards is limited, and the surface areas (on which adsorption can occur) of cellulose, lactose, and pectin are not as many as of activated carbon. Thus, cellulose, lactose, and pectin had less effective adsorption of the volatile standards.

Statistical Analysis

The experiment was designed as a three factor (volatile x adsorbent x day) experiment, which included 4 different volatile standards, 6 adsorbents, and 3 days, in a completely randomized model. Means, standard errors, sums of square, and mean squares were computed using the MSTATC microcomputer statistical program (Michigan State University, 1989).

Analysis of variance (ANOVA) of the relative concentration of the volatile standards was performed based on a three factor, split-plot, repeat-measure model. The ANOVA of the results (Table 3) showed significant interactions, therefore, conditional comparisons were required. In other words, under the condition of the same adsorbent and the same day, comparisons between any two of the volatile standards were necessary.Thus, for exampe on day 1, silica gel as the adsorbent, comparisons between any two of the volatiles (Table 4) had to be done in order to evaluate which volatile standard would be readily adsorbed by silica gel. With time (day) factor remaining the same (day 1), comparisons between any two of the volatile standards were repeated for activated carbon, sodium bisulfite, cellulose, lactose, and pectin independently. These procedures were repeated for day2 and day3 (Table 5,and 6). In the same way, comparisons between any two of the six adsorbents on

Table 3. Analysis of Variance for Relative % Concentration of Volatile Standards in the Vial Headspace

Source	Degree of Freedom	Sum of Squares	Mean Square	F Value
Factor A (Volatile Standards)	3	10048.009	3349.336	30.4868
Factor B (Adsorbents)	5	195515.242	39103.048	355.9200
AB	15	48740.349	3249.357	29.5767
Error (Bottle)	48	5273.371	109.862	
Factor C (Days)	2	17944.602	8972.301	285.9067
AC	6	6677.277	1112.880	35.4624
BC	10	2427.733	242.773	7.7361
ABC	30	2188.714	72.957	2.3248
Error	96	3012.664	31.382	
Total	215	291827.962		

Table 4. Comparisons of Volatile Standards for Each Adsorbent on Day1

Volatile Standards	Silica Gel	Activated Carbon	$\bar{y}_1 - \bar{y}_2$ Sodium Bisulfite	Cellulose	Lactose	Pectin
Butanal vs. Cycloheptatriene	1.516	0.393	68.922*	12.416	6.811	2.165
Butanal vs. Pentanal	1.745	0.393	64.527*	5.92	2.545	1.34
Butanal vs. Hexanal	56.375*	47.244*	42.711*	14.124	28.991*	10.337
Cycloheptatriene vs. Pentanal	0.229	0.000	4.395	6.496	4.266	0.825
Cycloheptatriene vs. Hexanal	57.891*	47.637*	26.211*	26.540*	35.802*	8.172
Pentanal vs. Hexanal	58.120*	47.637*	21.816*	20.044*	31.536*	8.997

*MSD= 16.674, any value larger than MSD would be considered significant difference.

**MSD calculation was shown in Appendix D.

Table 5. Comparisons of Volatile Standards for Each Adsorbnet on Day2

Volatile Standards	Silica Gel	Activated Carbon	$\bar{y}_1 - \bar{y}_2$ Sodium Bisulfite	Cellulose	Lactose	Pectin
Butanal vs. Cycloheptatriene	0.552	0.190	59.889*	17.061*	10.706	7.208
Butanal vs. Pentanal	0.660	0.190	57.764*	7.326	0.507	10.674
Butanal vs. Hexanal	22.485*	11.954	15.435	28.463*	36.016*	28.310*
Cycloheptatriene vs. Pentanal	0.108	0.000	2.215	9.735	11.213	3.646
Cycloheptatriene vs. Hexanal	23.037*	12.144	44.454*	45.524*	46.722*	21.282*
Pentanal vs. Hexanal	23.145*	12.144	42.329*	35.789*	35.509*	17.636*

*MSD= 16.674, any value larger than MSD would be considered significant difference.

**MSD calculation was shown in Appendix D.

Table 6. Comparison of Volatile Standards for Each Adsorbent on Day3

Volatile Standards	Silica Gel	Activated Carbon	$\overline{y_1} - \overline{y_2}$ Sodium Bisulfite	Cellulose	Lactose	Pectin
Butanal vs. Cycloheptatriene	0.022	0.095	69.334*	19.423*	7.908	0.218
Butanal vs. Pentanal	0.152	0.095	61.474*	3.031	10.978	9.361
Butanal vs. Hexanal	8.506	8.164	3.930	29.732*	41.331*	33.341*
Cycloheptatriene vs. Pentanal	0.130	0.000	7.860	16.392	18.886*	9.579
Cycloheptatriene vs. Hexanal	8.528	8.259	73.264*	49.155*	49.239*	33.559*
Pentanal vs. Hexanal	8.658	8.259	65.404*	32.763*	30.353*	23.980*

*MSD= 16.674, any value larger than MSD would be considered significant difference.

**MSD calculation was shown in Appendix D.

the same day for the same volatile standards were completed and are shown in Table 7, 8 and 9. A minimum significant difference (MSD) was computed (as shown in Appendix D) for five percent type I error. Any value of the comparisons larger than the MSD value was considered a significant difference.

Significant Difference of Conditional Comparisons of Volatile Standards for the Same Adsorbent

The conditional comparisons of any two volatile standards within each adsorbent at the same day (day 1, day 2, and day 3) are shown in Table 4, 5, and 6.

(1). Day 1

On day 1 with silica gel as the adsorbent, comparisons between any two of the volatile standards are shown in Table 4. There were three pairs of volatile standards having significant difference, these were butanal vs. hexanal, cycloheptatriene vs. hexanal, and pentanal vs. hexanal. Butanal, cycloheptatriene, and pentanal were adsorbed by silica gel more readily than hexanal, while there were no significant adsorption difference among butanal, cycloheptatriene, and pentanal (Figure 13).

With activated carbon as the adsorbent, comparisons of butanal vs. hexanal, cycloheptatriene vs. hexanal, and pentanal vs. hexanal were found to be significant different which meant that butanal, cycloheptatriene, and pentanal were adsorbed by activated carbon more easily than hexanal. With activated carbon as the adsorbent,

Table 7. Comparisons of Adsorbents for Each Volatile Standard on Day1

Adsorbents	$\bar{y}_1 - \bar{y}_2$ Butanal	Cycloheptatriene	Pentanal	Hexanal
Silica gel vs. Activated Carbon	1.352	0.229	0.000	10.483
Silica gel vs. Sodium Bisulfite	27.892*	98.330*	94.164*	14.228
Silica gel vs. Cellulose	75.411*	89.343*	83.076*	4.912
Silica gel vs. Lactose	89.262*	97.589*	93.552*	3.896
Silica gel vs. Pectin	95.544*	94.895*	95.949*	28.832*
Activated Carbon vs. Sodium Bisulfite	29.244*	98.559*	94.164*	24.711*
Activated Carbon vs. Cellulose	76.763*	89.572*	83.076*	15.395
Activatted Carbon vs. Lactose	90.614*	97.818*	93.552*	14.379
Activated Carbon vs. Pectin	96.896*	95.124*	95.949*	39.315*
Sodium Bisulfite vs. Cellulose	47.519*	8.987	13.088	9.316
Sodium Bisulfite vs. Lactose	61.370*	0.741	2.612	10.332
Sodium Bisulfite vs. Pectin	67.652*	3.435	0.215	14.604
Cellulose vs. Lactose	13.851	8.246	10.476	1.024
Cellulose vs. Pectin	20.133*	5.552	12.873	23.920*
Lactose vs. Pectin	6.282	2.694	2.397	24.936*

*MSD=18.625, any value larger than MSD would be considered significant difference.

**MSD calculation was shown in Appendix D.

Table 8. Comparisons of Adsorbents for Each Volatile Standard on Day2

Adsorbents	$\bar{y}_1 - \bar{y}_2$ Butanal	Cycloheptatriene	Pentanal	Hexanal
Silica gel vs. Activated Carbon	0.470	0.108	0.000	11.031
Silica gel vs. Sodium Bisulfite	20.512*	80.953*	78.936*	13.462
Silica gel vs. Cellulose	63.581*	81.194*	71.567*	12.633
Silica gel vs. Lactose	80.545*	91.803*	80.698*	22.044*
Silica gel vs. Pectin	90.178*	83.702*	80.164*	39.383*
Activated Carbon vs. Sodium Bisulfite	20.982*	81.061*	78.936*	24.493*
Activated Carbon vs. Cellulose	64.051*	81.302*	71.567*	23.664*
Activatted Carbon vs. Lactose	81.015*	91.911*	80.698*	33.075*
Activated Carbon vs. Pectin	90.648*	83.810*	80.164*	50.414*
Sodium Bisulfite vs. Cellulose	43.069*	0.241	7.369	0.829
Sodium Bisulfite vs. Lactose	60.033*	10.85	1.762	8.582
Sodium Bisulfite vs. Pectin	69.666*	2.749	1.228	25.921*
Cellulose vs. Lactose	16.964	10.609	9.131	9.411
Cellulose vs. Pectin	26.597*	2.508	8.597	26.750*
Lactose vs. Pectin	9.633	8.101	0.534	17.339

*MSD= 16.674, any value larger than MSD would be considered significant difference.

**MSD calculation was shown in Appendix D.

Table 9. Comparisons of Adsorbents for Each Volatile Standard on Day3

Adsorbents	Butanal	Cycloheptatriene	Pentanal	Hexanal
		$\bar{y}_1 - \bar{y}_2$		
Silica gel vs. Activated Carbon	0.057	0.130	0.000	0.399
Silica gel vs. Sodium Bisulfite	10.732	80.080*	72.358*	1.704
Silica gel vs. Cellulose	53.623*	73.068*	56.806*	15.385
Silica gel vs. Lactose	71.758*	79.688*	60.932*	21.921*
Silica gel vs. Pectin	76.310*	76.550*	67.101*	34.463*
Activated Carbon vs. Sodium Bisulfite	10.789	80.210*	72.358*	1.305
Activated Carbon vs. Cellulose	53.680*	73.198*	56.806*	15.784
Activatted Carbon vs. Lactose	71.815*	79.818*	60.932*	22.320*
Activated Carbon vs. Pectin	76.367*	76.680*	67.101*	34.862*
Sodium Bisulfite vs. Cellulose	42.891*	7.012	15.552	17.089
Sodium Bisulfite vs. Lactose	61.026*	0.392	11.426	23.625*
Sodium Bisulfite vs. Pectin	65.578*	3.530	5.257	36.167*
Cellulose vs. Lactose	18.135	6.620	4.126	6.536
Cellulose vs. Pectin	22.687*	3.482	10.295	19.078*
Lactose vs. Pectin	4.552	3.138	6.169	12.542

*MSD= 16.674, any value larger than MSD would be considered significant difference.

**MSD calculation was shown in Appendix D.

similar adsorption profiles were found for butanal, cycloheptatriene, and pentanal(Figure 14).

Only one comparison, cycloheptatriene vs. pentanal, showed no significant difference when sodium bisulfite was the adsorbent. As shown in Figure 15, sodium bisulfite exhibited substantial adsorption of butanal and hexanal, and had no significant adsorption of cycloheptatriene and pentanal.

For cellulose, the comparisons of cycloheptatriene vs. hexanal and pentanal vs. hexanal were significantly different. Cellulose had significant adsorption of hexanal in comparison to adsorption of cycloheptatriene and pentanal (Figure 16).

Butanal vs. hexanal, cycloheptatriene vs. hexanal, and pentanal vs. hexanal were observed to be significantly different with lactose as the adsorbent (Table 4). This indicated that more higher levels of hexanal were adsorbed than butanal, cycloheptatriene, and pentanal, and that there were no significant differences among butanal, cycloheptatriene, and pentanal (Figure 17).

There were no significant differences between any of the comparisons when pectin was the adsorbent (Table 4). In other words, the adsorption capacity of pectin was almost the same for butanal, cycloheptatriene, pentanal, and hexanal on day 1 (Figure 18).

(2). Day 2

Comparisons of the volatile standards adsorbed by silica gel showed that no significant differences were observed for butanal vs. hexanal, cycloheptatriene vs. hexanal, and pentanal vs. hexanal

(Table 5). Butanal, cycloheptatriene, and pentanal were adsorbed significantly by silica gel. Hexanal was also adsorbed by silica gel but less so than butanal, cycloheptatriene, and pentanal (Figure 13).

There were no significant difference observed in the comparisons of any two volatile standards with activated carbon as the adsorbent (Table 5, Figure 14).

The results of the comparisons of butanal vs. cycloheptatriene, butanal vs. pentanal, cycloheptatriene vs. hexanal, and pentanal vs. hexanal exhibited significant difference (Table 5), which revealed that sodium bisulfite had good adsorption of butanal and hexanal with less effective adsorption of cycloheptatriene and pentanal (Figure 15).

With cellulose as the adsorbent, the comparisons of butanal vs. cycloheptatriene, butanal vs. hexanal, cycloheptatriene vs. hexanal, and pentanal vs. hexanal had significant difference (Table 5). Cellulose had more adsorption of butanal than of cycloheptatriene, and adsorption of hexanal onto cellulose was significantly higher than adsorption of butanal, cycloheptatriene, or pentanal onto cellulose (Figure 16).

The amount of hexanal adsorbed by lactose was significantly more than the amounts of butanal, cycloheptatriene, or pentanal adsorbed (Table 5, Figure 17).

The comparisons of butanal vs. hexanal, cycloheptatriene vs. hexanal, and pentanal vs. hexanal were significantly different when pectin was the adsorbent (Table 5). This indicates that hexanal was adsorbed by pectin readily, while butanal, cycloheptatriene, and

pentanal showed no significant difference of adsorption by pectin (Figure 18).

(3). Day 3

The results of the comparisons of volatile standards for each adsorbent on day 3 are shown in Table 6.

There were no significant differences observed in the comparisons of volatile standards with silica gel as the adsorbent (Table 6). As shown in Figure 13, each volatile standard was highly adsorbed by silica gel, and thus no significant differences occurred.

No significant differences observed for the comparisons of volatile standards for activated carbon (Table 6). Each volatile standard was readily adsorbed by activated carbon (Figure 14).

With sodium bisulfite as the adsorbent, butanal vs. cycloheptatriene, butanal vs. pentanal, cycloheptatriene vs. hexanal, and pentanal vs. hexanal had significant difference (Table 6). Butanal and hexanal were adsorbed by sodium bisulfite while cycloheptatriene and pentanal were not (Figure 15).

The comparisons of volatile standards with cellulose as the adsorbent on day 3 had the same significances as on day 2. Hexanal was the most significantly adsorbed by cellulose (Figure 16). Butanal, cycloheptatriene, and pentanal had almost the same level of adsorption by cellulose.

In addition to the significant difference in the comparison of cycloheptatriene vs. pentanal, there was a significant different between hexanal and the other three volatile standards (butanal, cycloheptatriene, and pentanal) with lactose as the adsorbent (Table 6). Hexanal was the most highly adsorbed by lactose among the

volatile standards. The difference in adsorption by lactose between pentanal and cycloheptatriene was significant. Pentanal was adsorbed by lactose more than cycloheptatriene (Figure 17).

There was significant difference between butanal vs. hexanal, cycloheptatriene vs. hexanal, and pentanal vs. hexanal, with pectin as the adsorbent (Table 6). Hexanal was significantly adsorbed by pectin by comparing to the other volatile standards (butanal, cycloheptatriene, and pentanal) (Figure 18).

Significant Difference of Conditional Comparisons of Adsorbents for the Same Volatile Standard

Within the same day (day 1, day 2, and day 3), for each volatile standard (butanal, cycloheptatriene, pentanal, and hexanal), any two of six adsorbents (silica gel, activated carbon, sodium bisulfite, cellulose, lactose, and pectin) were paired for comparisons. A minimum significant difference (MSD) was computed as shown in Appendix D. Any value of the comparisons of the adsorbents larger than MSD was considered significantly different.

(1). Day 1

Comparisons for the adsorbents using butanal as the volatile standard are shown in Table 7. Comparisons between silica gel and four adsorbents (sodium bisulfite, cellulose, lactose, and pectin) were significantly different which means that silica gel had higher adsorption capacity of butanal than the other four adsorbents. Activated carbon had a similar adsorption capacity for butanal as did silica gel (Table 7). Sodium bisulfite also exhibited good adsorption of

butanal compared to adsorption by cellulose, lactose, and pectin (Figure 19).

Using cycloheptatriene as the volatile standard, the comparison results (Table 7) show significant difference for comparisons between silica gel and four adsorbents (sodium bisulfite, cellulose, lactose, and pectin). Activated carbon also had significant difference with the same four adsorbents (sodium bisulfite, cellulose, lactose, and pectin). Silica gel and activated carbon were the most effective adsorbents of cycloheptatriene (Figure 20).

The effectiveness of the adsorbents on pentanal were the same as previously for cycloheptatriene. Silica gel and activated carbon were the most effective adsorbents for pentanal (Figure 21).

Results were significantly different (Table 7) for comparisons of the adsorbents with hexanal as the volatile standard. Pectin was the least effective adsorbent of hexanal. Silica gel, cellulose, and lactose had similar adsorption profiles for hexanal on day 1, while activated carbon exhibited the greatest adsorption of hexanal (Figure 22).

(2). Day 2

As shown in Table 8, the comparisons for the adsorption capacity of the adsorbents with butanal was essentically the same as on day 1. Silica gel and activated carbon adsorbed almost all of the butanal in the vial headspace. Sodium bisulfite had significant adsorption of butanal compared to cellulose, lactose, and pectin (Figure 19).

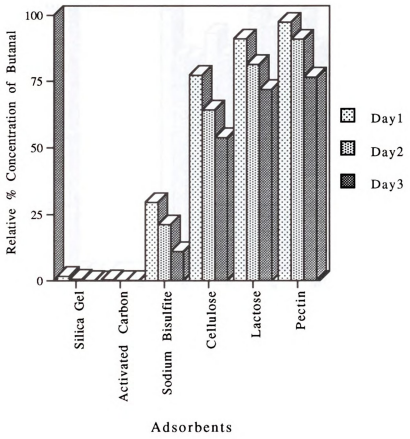


Figure 19. Relative % Concentration of Butanal Remaining in the Vial Headspace

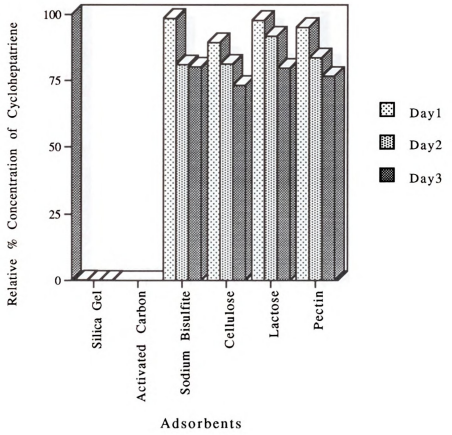


Figure 20. Relative % Concentration of Cycloheptatriene Remaining in the Vial Headspace

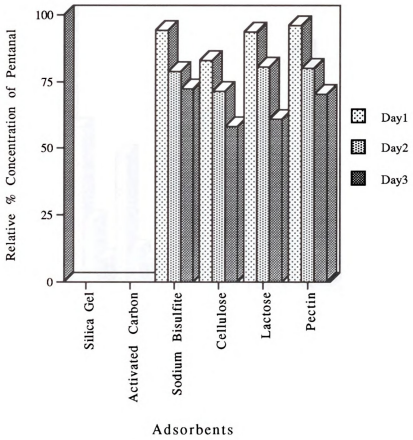


Figure 21. Relative % Concentration of Pentanal Remaining in the Vial Headspace

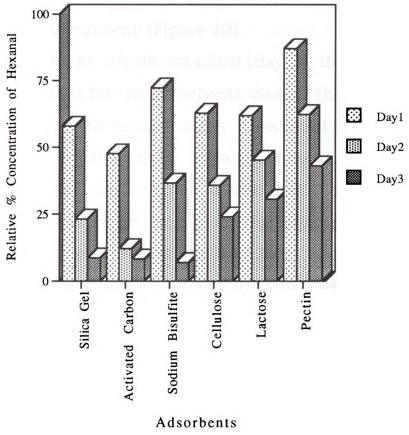


Figure 22. Relative % Concentration of Hexanal Remaining in the Vial Headspace

For cycloheptatriene, significant difference was observed in the comparisons of silica gel to sodium bisulfite, cellulose, lactose, and pectin. Similarly comparison between activated carbon and sodium bisulfite, cellulose, lactose, and pectin were significantly different (Table 8). Silica gel and activated carbon were the most significant adsorbents for cycloheptatriene (Figure 20).

With pentanal as the volatile standard (day 2), the results (Table 8) of comparisons for the adsorbents showed the same level of significant difference as the results on day 1. Pentanal was not detectable in the vial headspace with silica gel or activated carbon as the adsorbent, while the relative % concentration of pentanal in the vial headspace remained high when sodium bisulfite, cellulose, lactose, or pectin was the adsorbent (Figure 21).

On day 2, the adsorption capacity of the adsorbents with hexanal had changed from day 1. The adsorption capacity of silica gel vs. lactose and pectin were significantly different. The amount of hexanal adsorbed by silica gel was higher than the amount of hexanal adsorbed by lactose or pectin. The comparisons between activated carbon and sodium bisulfite, cellulose, lactose, and pectin were significantly different, which meant activated carbon offered the greatest adsorption of hexanal (Figure 22).

(3). Day 3

The significant difference in the comparisons of adsorbents with butanal as the volatile standard (day 3) are shown in Table 9. The comparisons between silica gel and three adsorbents (cellulose, lactose, and pectin) were significantly different which meant that

silica gel had a higher adsorption capacity for butanal than the other three adsorbents. Activated carbon had similar adsorption of butanal as did silica gel (Table 9). Sodium bisulfite also exhibited good adsorption of butanal compared to the adsorption of cellulose, lactose, and pectin (Figure 19).

Using cycloheptatriene as the volatile standard, the comparison results (Table 9) show significant difference for comparisons between silica gel and sodium bisulfite, cellulose, lactose, and pectin. Activated carbon also had significant difference with sodium bisulfite, cellulose, lactose, and pectin. Silica gel and activated carbon had the most effective adsorption of cycloheptatriene among the adsorbents (Figure 20).

On day 3, the effectiveness of the adsorbents with pentanal was the same as previously discussed with cycloheptatriene. Silica gel vs. sodium bisulfite, cellulose, lactose, and pectin were significantly different. Activated carbon vs. sodium bisulfite, cellulose, lactose, and pectin were also significantly different (Table 9). Silica gel and activated carbon could adsorb pentanal more effectively than the other four adsorbents (Figure 21).

Comparisons of the adsorbents with hexanal are shown in Table 9. Silica gel vs. lactose, and pectin were significantly different. Activated carbon vs. lactose, and pectin exhibited significant difference. Sodium bisulfite vs. lactose, and pectin were significantly different. Significant difference was also observed in the comparison of cellulose vs. pectin. Silica gel, activated carbon, and sodium bisulfite adsorbed hexanal similarly and which were better than

lactose and pectin. The effectiveness of cellulose with hexanal was better than pectin (Figure 22).

Overall, both silica gel and activated carbon exhibited significant adsorption of butanal, cycloheptatriene, and pentanal in comparison to the other four adsorbents (sodium bisulfite, cellulose, lactose, and pectin) on day 1, 2 and 3.

Effect of Selected Adsorbents on Oxidized Frozen Lake Trout

Some investigators have found that volatile aldehydes contributed strongly to the characteristic aroma of oxidized fish lipids (Lea, 1953; Yu et al., 1961; Aiken and Connell, 1979; Ikeda, 1980). In this study, volatile aldehydes such as butanal, pentanal, and hexanal were found as the major off-flavor compounds in oxidized frozen lake trout.

Low temperature storage (Young, 1950; Dyer and Morton, 1956; Peters and McLane, 1959), application of antioxidant (Brown et al., 1957; Andersson and Danielson, 1961; Yu et al., 1969; Sweet, 1973; Love and Pearson, 1974; Deng et al., 1977), and packaging technique (Griffin, 1980; Gorga, 1988; Connell, 1990) etc. can be used to prevent off-flavor development in fish during storage. Shelf life is extended by lowering the storage temperature (Peters and McLane, 1959). However, temperature fluctuation can cause quality deterioration, an increase in degree of lipid oxidation, and shorten the storage life of frozen fish (Dyer, 1959; Lentz and Rooke, 1960; Palmateer et al., 1960; Lane, 1966). Application of phenolic antioxidants in frozen fish fillets was found not effective in retarding oxidative rancidity (Stuckey, 1968). Inadequate distribution of the

antioxidants may limit the usage on fish samples (Sweet, 1973). Gas impermeable packaging needs to be associated with low temperature (below 4°C) storage, otherwise it may cause an outgrowth of *Clostridium botulinum* (Gorga, 1988). Therefore, a different approach was used in this research to reduce concentration of off-flavor compounds in oxidized frozen lake trout using adsorbents to adsorb the off-flavor compounds.

The adsorbing capacity of six adsorbents was evaluated, and activated carbon and silica gel were found to have the greatest capacity.

The capability of activated carbon and silica gel in reducing off-flavor development in oxidized frozen lake trout was determined.

Depositing 40 grams of fish sample (frozen lake trout) in a 450 ml glass jar with a screw cap which has a sampling port on the top as shown in Figure 4 . Two grams adsorbent, silica gel or activated carbon, was packed in a Tyvek pouch (1.0 in x 1.0 in) and suspended above the fish sample and stored at 5°C, for three days. Analysis of the headspace in the jar was completed by GC. The most abundant volatile (retention time: 4.70 min.) was employed to monitor the adsorption activity of activated carbon and silica gel. The area response analyses of the most abundant volatile are shown in Table 10. The area response of the control was designated 100% relative concentration of volatile. Using this as the baseline, Table 10 was converted into Table 11.

Table 10. Area Response[1] of Volatile[2] in the Headspace of Fish Samples after 72 Hours Storage at 5°C

	Fish Sample		
Adsorbent	1	2	3
Silica gel	307515.0	3808.5	29545.0
Activated Carbon	3167.0	2040.5	339.5
Control	315675.0	5776.0	25100.5

1. All values represent the average of two replicated experiments.
2. The most abundant volatile (retention time: 4.70 min.)

Table 11. Relative % Concentration[1] of Volatile[2] in the Headspace of Fish Samples after 72 Hours Storage at 5°C

	Fish Sample		
Adsorbent	1	2	3
Silica gel	97.41 ± 5.14	65.94 ± 3.49	99.99 ± 1.74
Activated Carbon	1.00 ± 0.40	34.33 ± 2.08	1.35 ± 0.22

1. All values represent the average of two replicated experiments.
2. The most abundant volatile (retention time: 4.70 min.)

Adsorption Activity of Activated Carbon for Frozen Lake Trout

With activated carbon as the adsorbent, the relative % concentrations of the most abundant volatile in the jar headspace (Table 11) were 1.00%, 34.33%, and 1.35% for fish sample 1, 2, and 3 respectively after 72 hours storage at 5°C. This indicates that activated carbon has the capability of adsorbing off-flavor volatiles and is compatible with the findings of other researchers (Tausig and Drake, 1959; Gernon et al., 1961; Brooks and Morr, 1982; Ruthven, 1984; Sakaki et al., 1984; Toro-Vazquez et al., 1991).

The adsorption activity of activated carbon for fish sample 2 (34.33%) was less effective than for fish sample 1 (1.00%), and 3 (1.35%). This variation in results could be due to experimental error.

Adsorption Activity of Silica Gel for Frozen Lake Trout

Using silica gel as the adsorbent, the relative % concentration of the most abundant volatile in the jar headspace (Table 11) were 97.41%, 65.94%, and 99.99% for fish sample 1, 2, and 3 respectively after 72 hours storage at 5°C. This reveals that silica gel had poor adsorption activity for the off-flavor volatiles from frozen lake trout. Some investigators reported that silica gel had good adsorption capacity for aromatics, alcohols, phenols, amines, hydrocarbons (Davis et al., 1952; Ruthven, 1984; Chou et al., 1986; Ackman and Ratnayake, 1989b; Shantha and Ackman, 1991). This variation could have been caused by the moisture in the fish which may have competed with the volatiles for surface adsorption. The presence of hydroxyl groups in silica gel imparts a degree of polarity to the surface, therefore water vapor can be adsorbed readily (Ruthven,

1984). The high capacity of silica gel to adsorb relatively large quantities of water was known in the early work on silica gel and this high adsorptive capacity was explained on the basis of muti-layer adsorption (Scott, 1993). While, the adsorption of water was greatly influenced by the hydrophobicity of the external surface and the micropores of actived carbon. This indicated that he hydrogen-bonded structure, by which water is generally adsorbed, was not viable within the constricted environment of the hydrophobic micropore of activated carbon (Bansal et al., 1988). Thus, the adsorption capacity of activated carbon has not been affected by the moisutre remaining in the fish.

Sensory Evaluation

A paired comparisons test was used for sensory evaluation. Six panelists were asked to sniff the fish samples (frozen lake trout) with activated carbon (treatment) and with glass beads (control) after storage at 5^{o}C for 3 days. Each panelist was asked to indicate which sample had the stronger off-odor. This sniff-test was repeated three times by the same group of panelists on different dates. The corresponding sensory scores are presented in Table 12.

Five judges chose the control (with glass beads) as stronger at the first session. All judges indicated that the control had the stronger off-odor at the second session, and five judges gave right verdicts at the third session (Table 12). 18 judgments were made after three sessions, and 16 out of 18 were correct judgments. According to Larmond (1977), in a two-sample difference test one sample must be selected 13 times, 15 times, and 16 times out of 18 judgments to be

significantly different at the 5%, 1%, and 0.1% level of significance respectively. In the sensory score, 16 times out of 18 judgments were right verdicts which was significantly different at the 0.1% level of significance. There was detectable difference in off-odor between the two treatments.

The results of sensory evaluation confirmed that the adsorption activity of activated carbon was significantly effective in decreasing off-flavor volatiles in oxidized frozen lake trout.

Table 12. Sensory Score of Frozen Lake Trout With Activated Carbon in a Jar after Storage at 5°C for 3 Days

Judge	1st Time	2nd Time	3rd Time
1	X	R	R
2	R	R	R
3	R	R	R
4	R	R	X
5	R	R	R
6	R	R	R
Total	5R	6R	5R

X=Wrong verdict

R=Right verdict

16 out of 18 correct judgments

SUMMARY AND CONCLUSIONS

Identification of the major off-flavor volatiles in frozen lake trout was completed by GC/MS. Butanal, cycloheptatriene, pentanal, and hexanal were identified as the major off-flavor compounds from the oxidized frozen lake trout by matching the GC/MS spectrum with the Mass spectrum standards. The adsorption of six adsorbents on four volatile standards was examined. Evaluation of the adsorbents with frozen lake trout was carried out to determine the efficiency of the adsorbents to control off-flavor of the frozen lake trout during storage.

In experiment II, silica gel and activated carbon exhibited significant adsorption effects on the volatile standards which were butanal, cycloheptatriene, pentanal, and hexanal. Therefore, both silica gel and activated carbon were used as adsorbents in further studies.

Silica gel and activated carbon were included in jars containing the frozen fish samples. Silica gel was ineffective in adsorbing the off-flavor volatiles probably due to adsorption of the moisture, while activated carbon suppression the off-flavor volatiles. Sensory evaluation also demonstrated the effectiveness of activated carbon. The results showed that activated carbon has potential as an adsorbent to control off-flavor of frozen fish during storage. Application of activated carbon to food products needs verification.

Eliminating the moisture adsorption by silica gel may allow it to be used. Sodium bisulfite has great adsorption of particular volatiles; cellulose, lactose, and pectin have adsorption of some volatiles. Their adsorption capacity may not be as good as activated carbon, they may be suitable for particular purpose of other adsorption.

APPENDIX A

APPENDIX A

GC Mass Spectrometry Analysis

47 peaks were recorded for the identification of volatile aroma compounds from oxidized frozen lake trout (Figure A-1). Four peaks (retention time: 3.912, 4.070, 8.254, and 27.020 min.) were in the most abundant quantity which were represented in area response (Table A-1). The mass spectrum of these four volatile compounds were shown in Figure A-2, A-3, A-4, and A-5. Butanal, pentanal, cycloheptatriene, and hexanal were identified as these four volatile compounds based on coincidence of mass spectral patterns of standards.

Figure A-1. Mass Chromatogram of Volatile Aroma Compounds from Oxidized Frozen Lake Trout

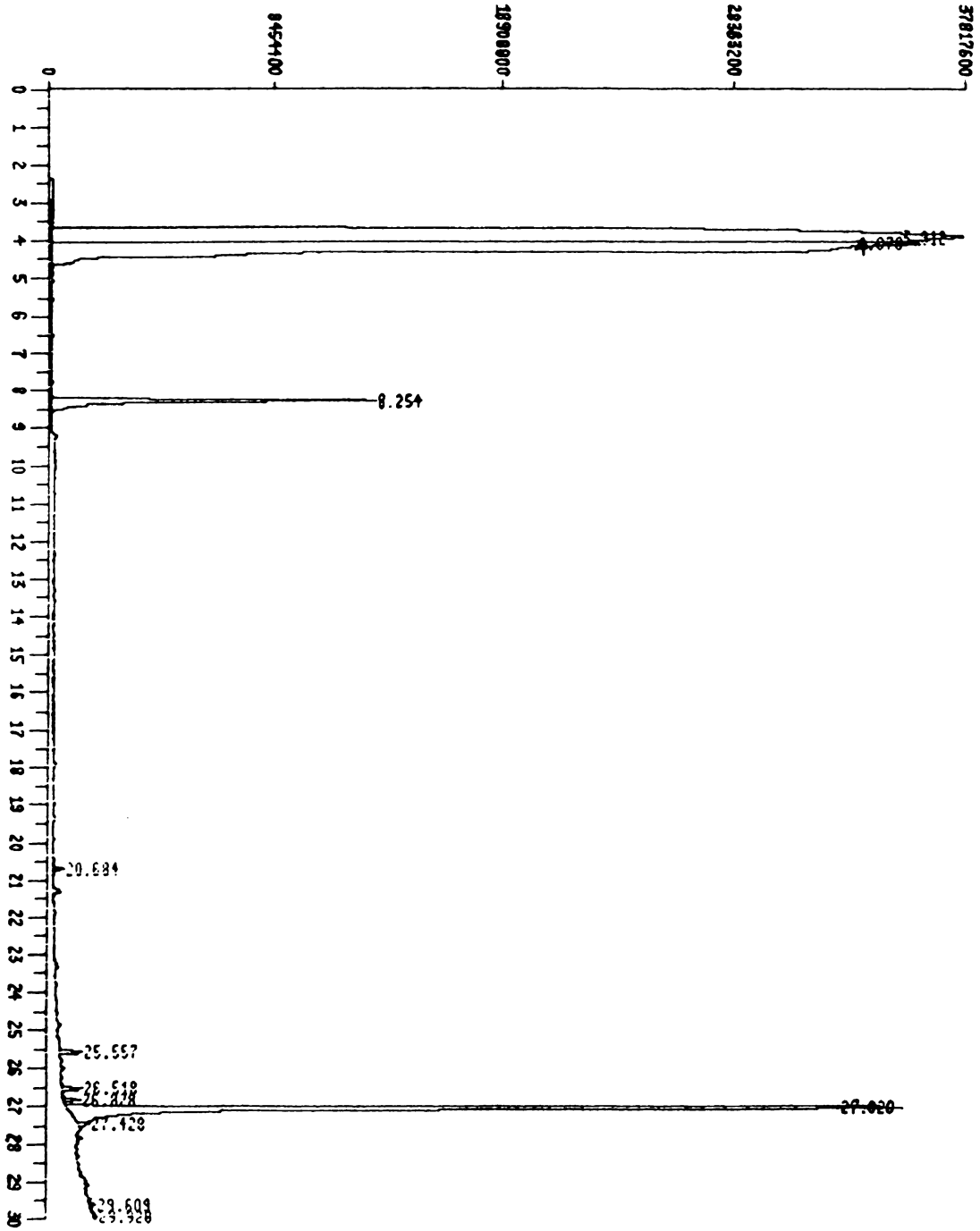


Table A-1. Retention Time, Area Response, of Volatile Aroma Compounds from Oxidized Frozen Lake Trout

Peak#	Ret Time	Type	Width	Area	Start Time	End Time
1	3.912	BV	0.256	7582772058	3.613	4.036
2	4.070	VB	0.301	6468088100	4.036	4.757
3	8.254	BV	0.088	786325963	8.116	8.623
4	20.684	PV	0.070	21684032	20.522	20.778
5	25.557	PV	0.054	37359175	25.409	25.661
6	26.518	BV	0.054	29620344	26.323	26.602
7	26.828	BV	0.072	26627679	26.667	26.893
8	27.020	PV	0.106	2384072632	26.893	27.411
9	27.428	VV	0.085	25953732	27.411	27.572
10	29.609	VV	0.068	13781100	29.524	29.739
11	29.928	PV	0.103	12059580	29.739	30.001
12	30.334	VV	0.059	35382387	30.221	30.426
13	30.537	VV	0.053	14201909	30.467	30.571
14	30.608	VV	0.049	20810458	30.571	30.661
15	30.728	VV	0.069	21659085	30.661	30.793
16	30.996	VV	0.126	28889195	30.793	31.012
17	31.043	VV	0.045	8598594	31.012	31.064
18	31.140	VV	0.072	22958538	31.064	31.179
19	31.231	VV	0.097	24018807	31.179	31.292
20	31.418	VV	0.111	32150752	31.292	31.433
21	31.498	VV	0.089	33415050	31.433	31.545
22	31.668	VV	0.111	55797155	31.545	31.701
23	31.817	VV	0.080	103466106	31.701	31.884
24	32.210	VV	0.216	201578915	31.884	32.240
25	32.366	VV	0.142	158436126	32.240	32.446
26	32.467	VV	0.037	28203892	32.446	32.484
27	32.516	VV	0.049	47117479	32.484	32.550
28	32.616	VV	0.080	72247295	32.550	32.652
29	32.753	VV	0.108	113078637	32.652	32.801
30	32.887	VV	0.089	83614048	32.801	32.911
31	33.008	VV	0.069	233068950	32.911	33.084
32	33.115	VV	0.046	36919602	33.084	33.131
33	33.366	VV	0.149	175481277	33.197	33.412
34	33.491	VV	0.093	75958041	33.412	33.508
35	33.649	VV	0.118	189323347	33.508	33.698
36	33.724	VV	0.099	88411237	33.698	33.803
37	34.008	VV	0.100	110747168	33.927	34.062
38	34.186	VV	0.066	370458178	34.062	34.262
39	34.308	VV	0.084	92308397	34.262	34.384
40	35.390	VV	0.067	437260182	35.209	35.498
41	35.653	VV	0.108	81511276	35.580	35.755
42	36.202	VV	0.089	85367652	36.098	36.289
43	36.668	VV	0.065	285247500	36.498	36.775
44	37.867	VV	0.077	171092343	37.733	37.974
45	38.099	VV	0.065	198027732	37.974	38.221
46	39.755	VV	0.071	110700992	39.634	39.890
47	41.722	BV	0.073	47239001	41.638	41.836

Figure A-2. Mass Spectrum of Peak #1 (Retention Time: 3.912)

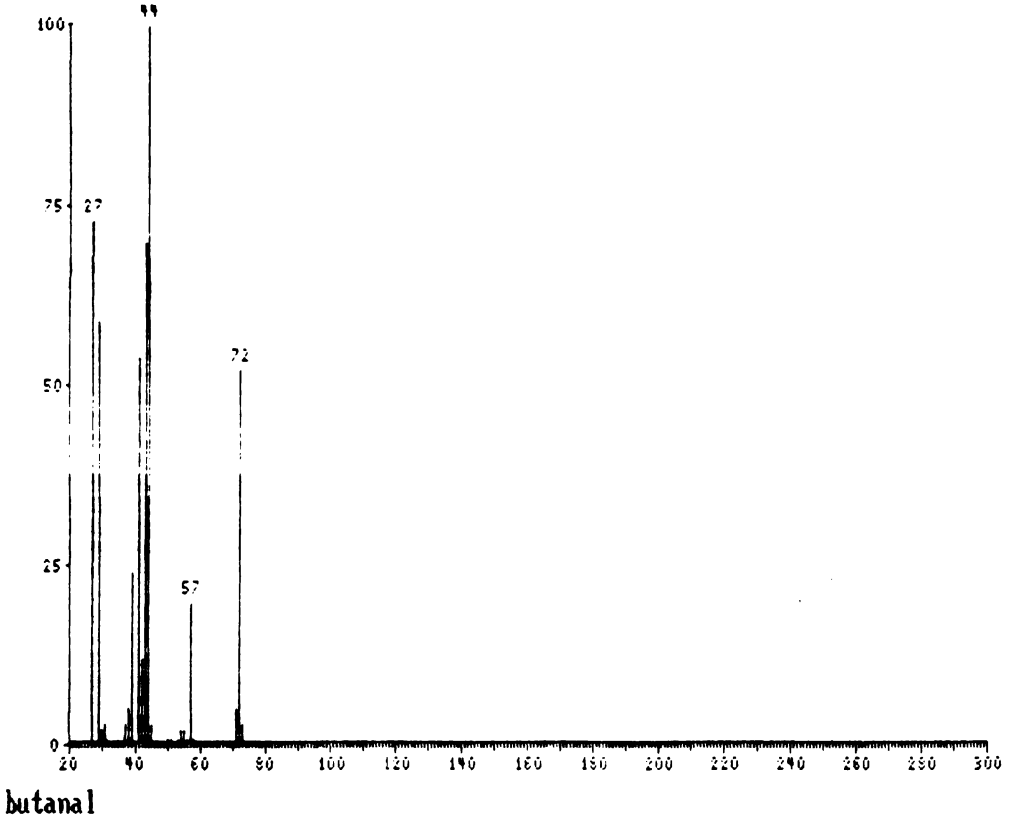
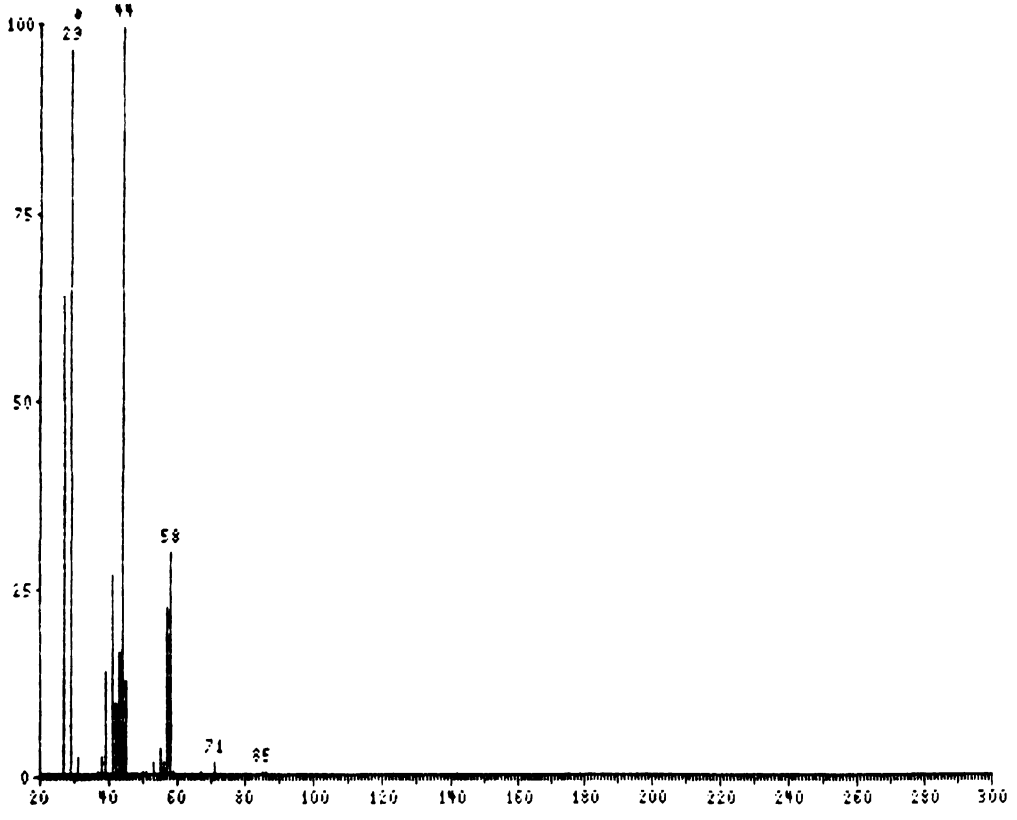


Figure A-3. Mass Spectrum of Peak #2 (Retention Time: 4.070)



pentanal

Figure A-4. Mass Spectrum of Peak #3 (Retention Time: 8.254)

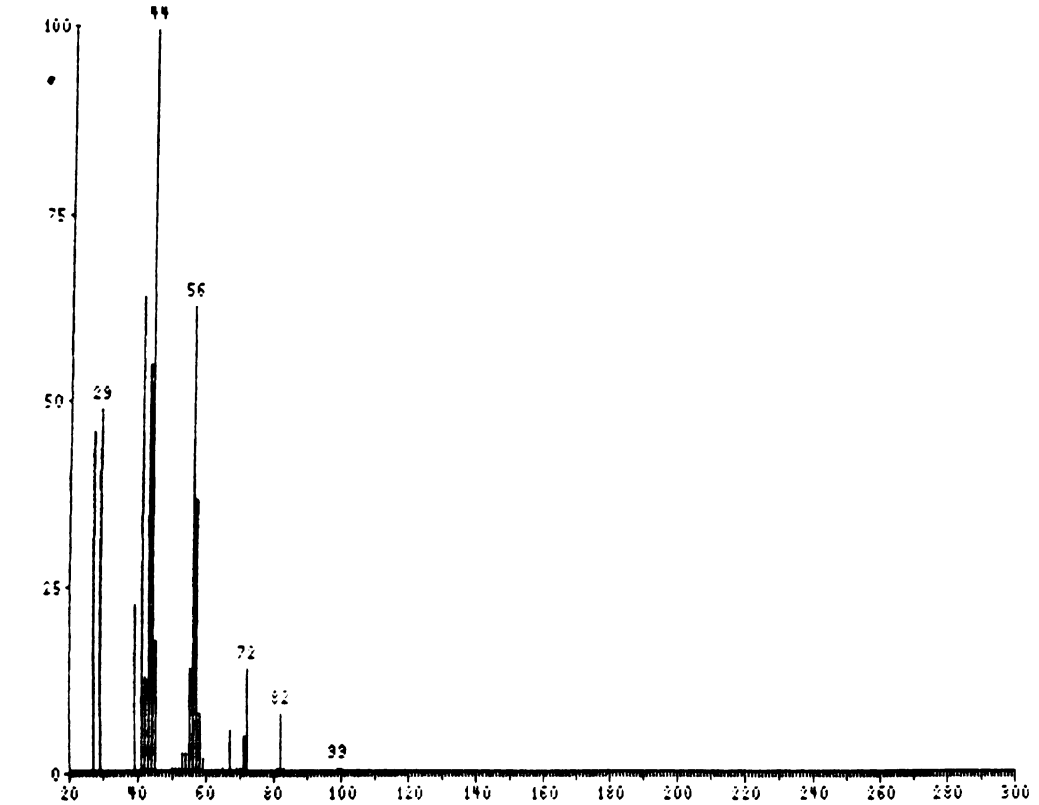
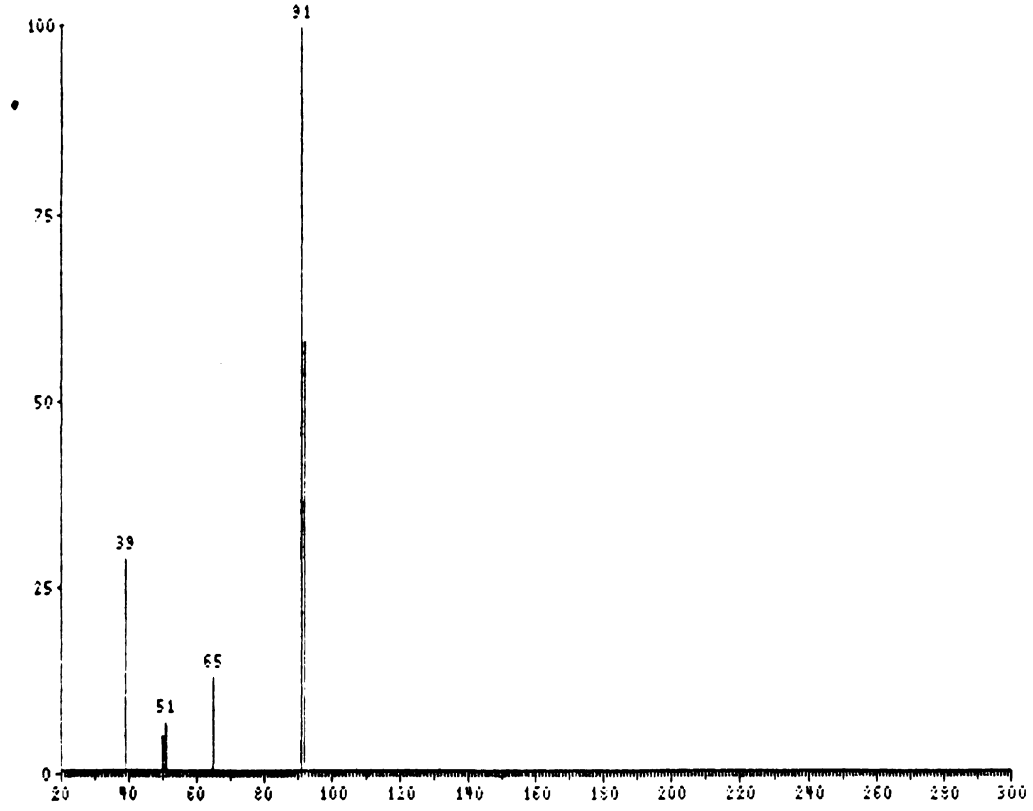


Figure A-5. Mass Spectrum of Peak # 8 (Retention Time: 27.020)



1,3,5-Cycloheptatriene

APPENDIX B

APPENDIX B

Standard Calibration

Standard calibration curves for butanal, cycloheptatriene, pentanal, and hexanal were established. Five concentrations:10ppm, 100ppm, 500ppm, 1000ppm, and 2000ppm(Vol./Vol.) were prepared for each volatile standard in o-dichlorobenzene as the solvent. In order to generate the data for the calibration curves, 0.5 μ l of sample was injected directly into the gas chromatography every 24 hours up to 72 hours. The area response was recorded under the following conditions.

Column: Supelcowax10: 0.25mm i.d. x 60mm capillary column

Conditions:

Range: 4

Helium carrier gas: 27ml/min

Column temperature: 150°C

Detector temperature: 250°C

Injection temperature: 220°C

The calibration data and the standard calibration curves for butanal were shown in Table B-1, Figure B-1, B-2, and B-3.

For cycloheptatriene, the calibration data and the standard calibration curves were shown in Table B-2, Figure B-4, B-5, and B-6.

The calibration data and the standard calibration curves for pentanal were shown in Table B-3, Figure B-7, B-8, and B-9.

For hexanal, the calibration data and the standard calibration curves were shown in Table B-4, Figure B-10, B-11, and B-12.

Table B-1 Butanal Calibration Data

Concentration (ppm, V/V)	Quantity of butanal Injected X10^{-6}(g)	Average Area Response* Day1	Day2	Day3
10	0.004	1343	1403	1292
100	0.04	20380	18855	31167
500	0.2	82765	89079	93650
1000	0.4	163680	232110	188710
2000	0.8	327270	356720	348030

*Retention time: 2.08min

Density: 0.80g/ml

Table B-2 Cycloheptatriene Calibration Data

Concentration (ppm, V/V)	Quantity of cyclohe. Injected X10^{-6}(g)	Average Area Response* Day1	Day2	Day3
10	0.00444	4600	3692	4532
100	0.0444	44158	44126	43054
500	0.222	212050	203250	190180
1000	0.444	473640	408900	397060
2000	0.888	924540	789660	965090

*Retention time: 4.55min.

Density: 0.888g/ml

Table B-3 Pentanal Calibration Data

Concentration (ppm, V/V)	Quantity of Pentanal Injected X10^{-6}(g)	Average Area Response* Day1	Day2	Day3
10	0.00405	1809	2070	2401
100	0.0405	21975	20975	20155
500	0.2025	88543	86873	77022
1000	0.405	213780	230310	207470
2000	0.810	448480	455310	367150

*Retention time: 3.09min.

Density: 0.810g/m

Table B-4 Hexanal Calibration Data

Concentration (ppm, V/V)	Quantity of Hexanal Injected X10^{-6}(g)	Average Area Response* Day1	Day2	Day3
10	0.00417	3707	3497	3534
100	0.0417	47129	37585	39639
500	0.2085	213610	202410	178010
1000	0.417	336830	316380	361080
2000	0.834	669880	649100	781880

*Retention time: 5.32min.

Density: 0.834g/ml

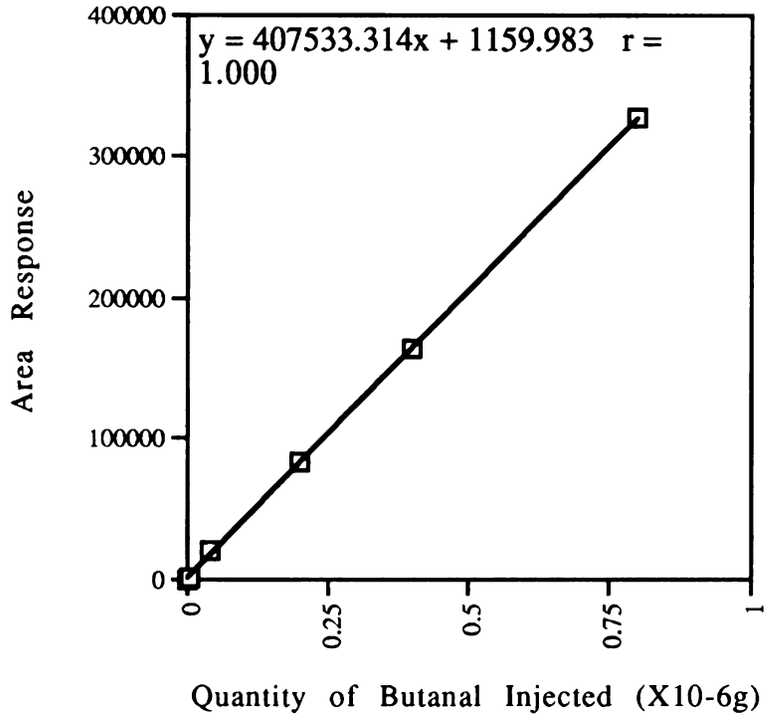


Figure B-1. Butanal Calibration Curve on Day1

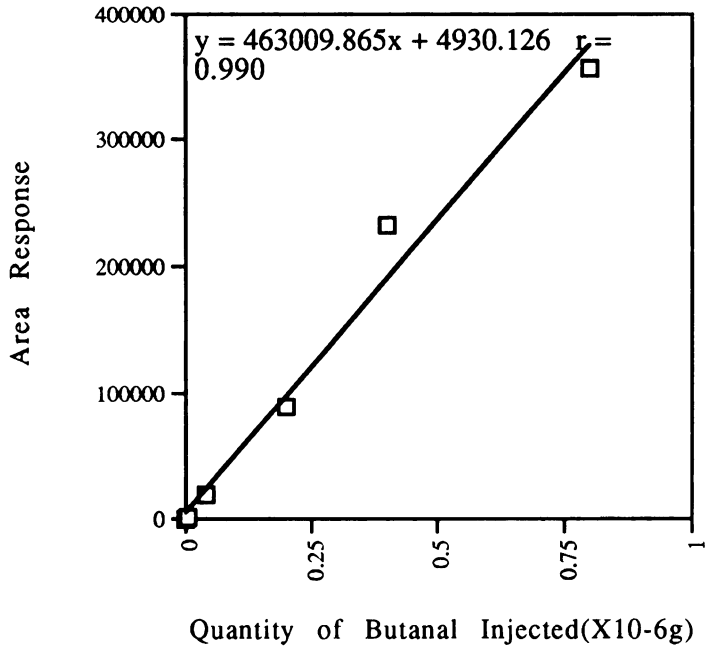


Figure B-2. Butanal Calibration Curve on Day2

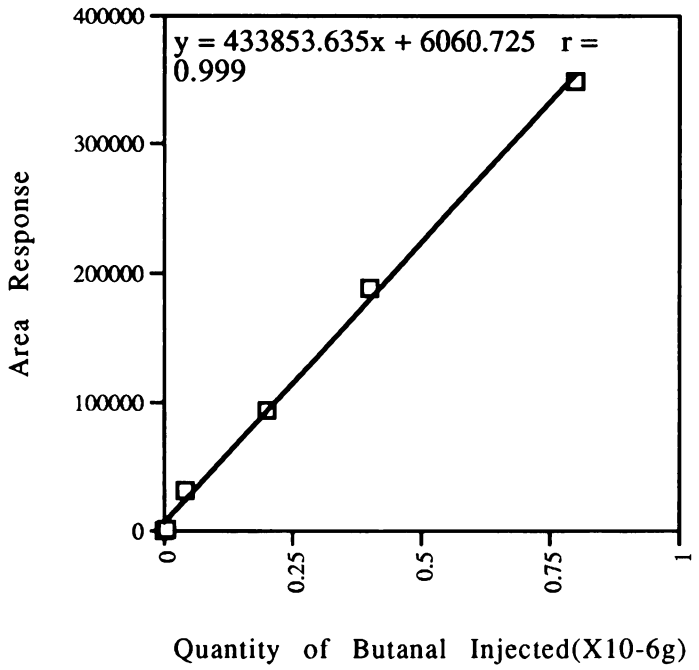


Figure B-3. Butanal Calibration Curve on Day3

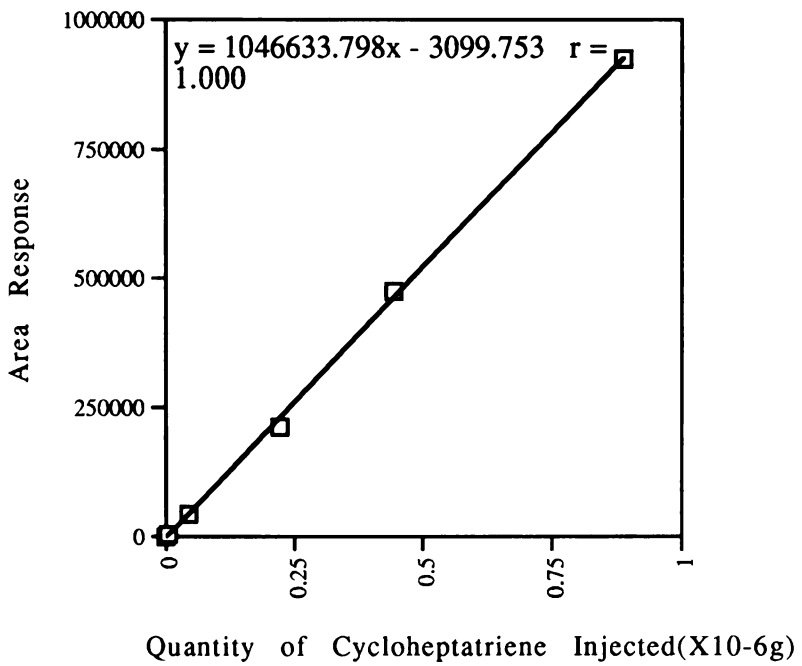


Figure B-4. Cycloheptatriene Calibration Curve on Day 1

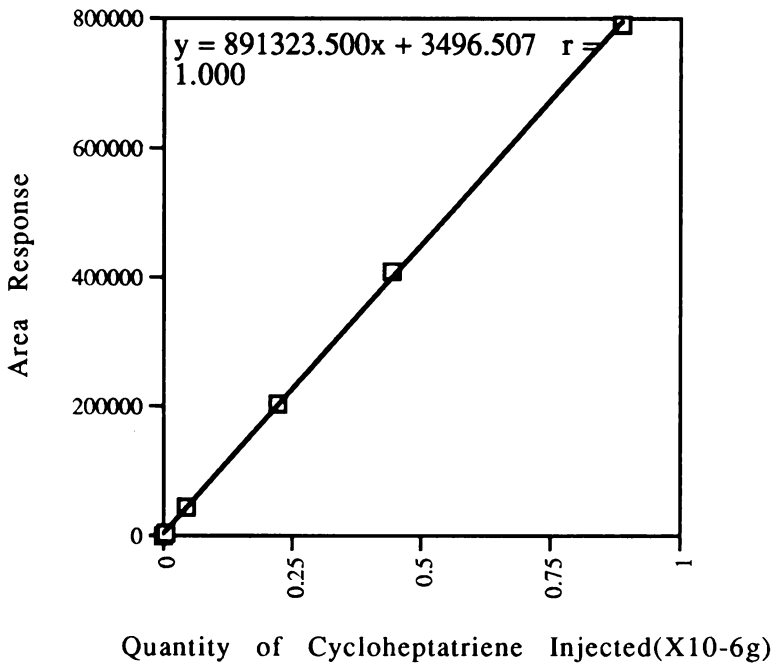


Figure B-5. Cycloheptatriene Calibration Curve on Day2

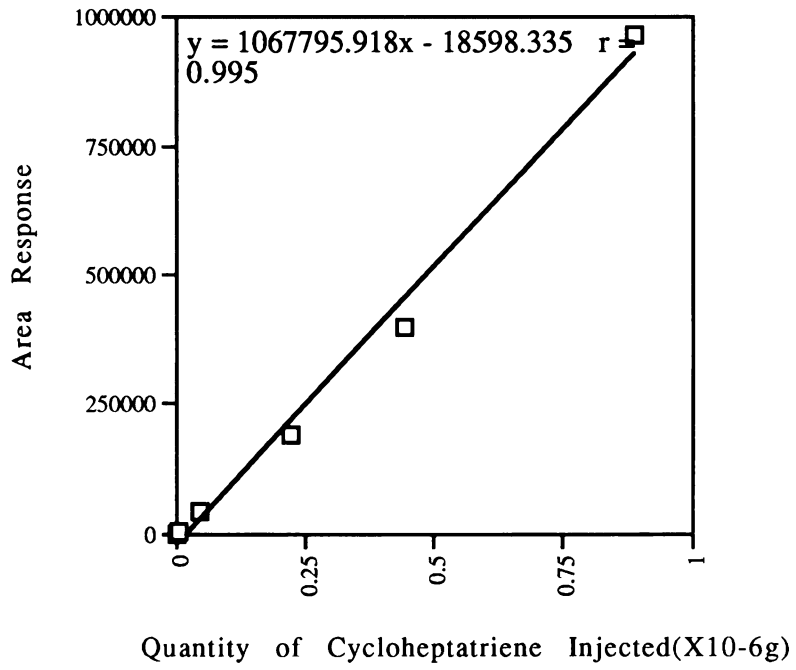


Figure B-6. Cycloheptatriene Calibration Curve on Day3

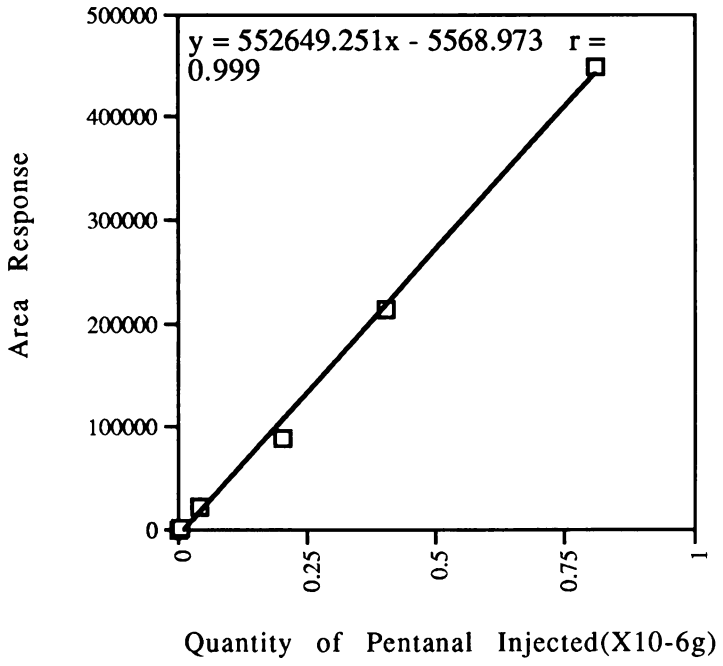


Figure B-7. Pentanal Calibration Curve on Day1

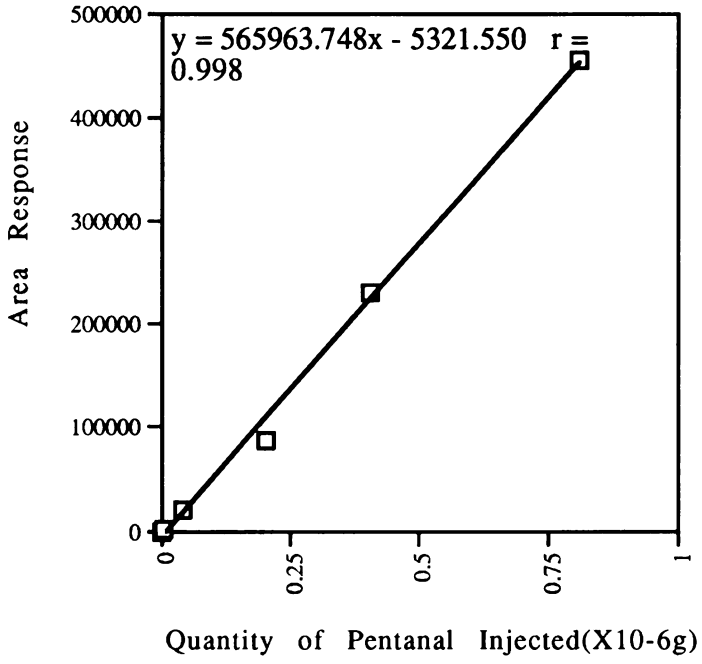


Figure B-8. Pentanal Calibration Curve on Day2

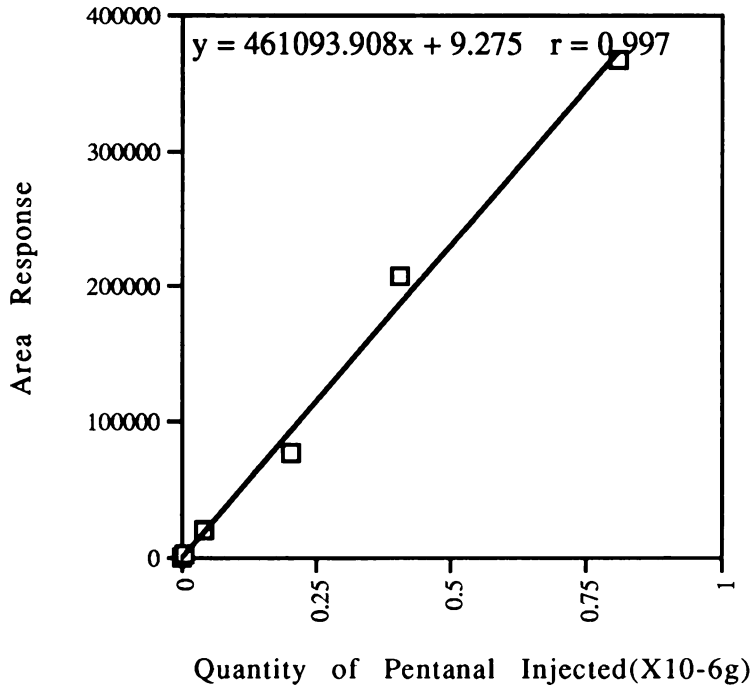


Figure B-9. Pentanal Calibration Curve on Day3

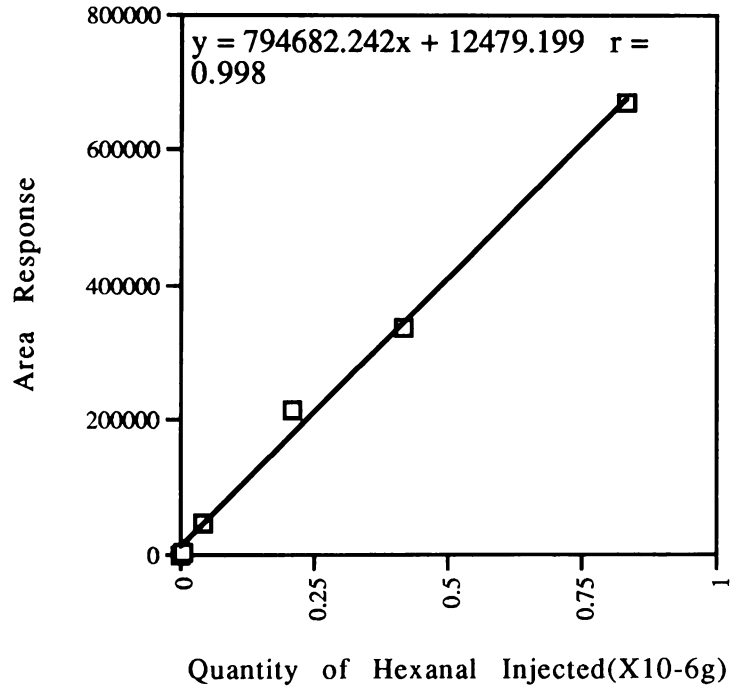


Figure B-10. Hexanal Calibration Curve on Day1

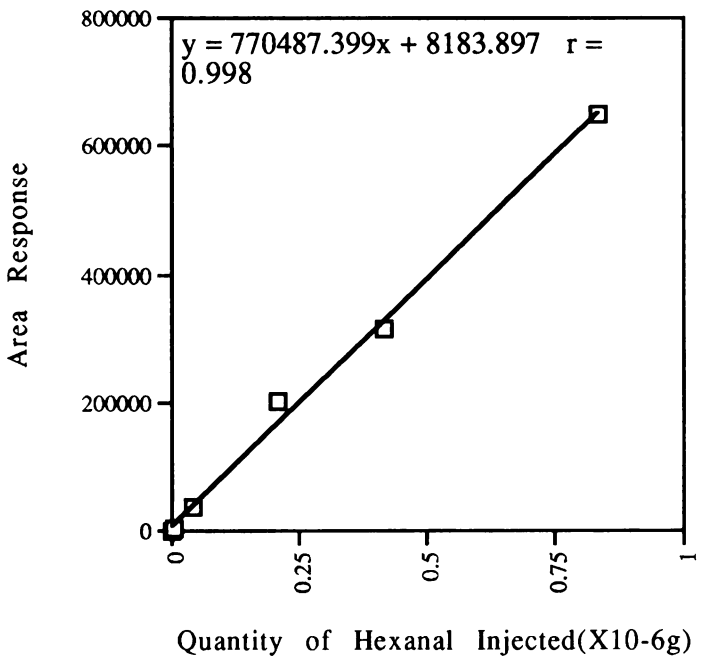


Figure B-11. Hexanal Calibration Curve on Day2

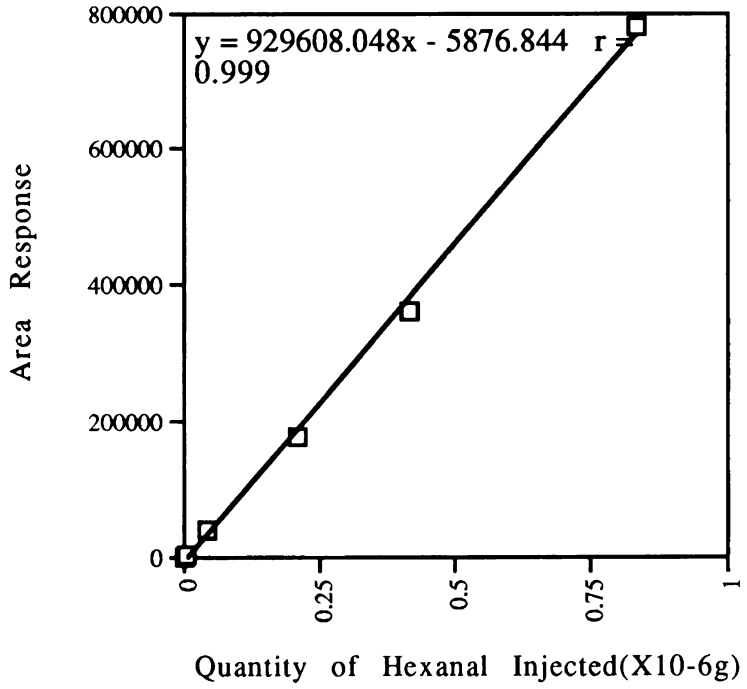


Figure B-12. Hexanal Calibration Curve on Day3

APPENDIX C

APPENDIX C

Original Experimental GC Data

The original experimental GC data were basically area responses recorded at particular retention times for the volatile standards.

The area responses recorded for butanal, cycloheptatriene, pentanal, and hexanal after 3 days storage, were respectively presented in Table C-1, C-2, C-3, and C-4.

Data were transformed from area responses to relative % concentration and shown in Table C-5, C-6, C-7, and C-8.

Table C-1. Area Response of Butanal in Cooperation with Adsorbents after 3 Days Storage

Adsorbents	Day1	Day2	Day3
Silica Gel	1612	384	ND
	305	ND	ND
	9058	3767	953
Activated	346	ND	ND
Carbon	1921	1193	599
	206	ND	ND
Sodium	73278	47932	23024
Bisulfite	30220	2221	330
	82895	82959	45097
Cellulose	140450	112820	98827
	160020	128330	108780
	184770	162870	130590
Lactose	180700	159330	133130
	190785	170235	150750
	200870	181140	168370
Pectin	209470	192470	156640
	194320	197940	176300
	208070	180880	147940

*All samples were three replications.

Table C-2. Area Response of Cycloheptatriene in Cooperation with Adsorbents after 3 Days Storage

Adsorbents	Day1	Day2	Day3
Silica Gel	1346	802	982
	686	598	ND
	748	502	ND
Activated	ND	ND	ND
Carbon	ND	ND	ND
	ND	ND	ND
Sodium	394350	368710	362780
Bisulfite	402720	280330	326920
	396970	333000	282140
Cellulose	368530	357970	333130
	381020	328325	286060
	335620	298680	267600
Lactose	382500	385680	346560
	403700	342830	309920
	398860	384990	310510
Pectin	373670	337800	318830
	400660	350980	332460
	378100	326580	277690

*All samples were three replications.

Table C-3. Area Response of Pentanal in Cooperation with Adsorbents after 3 Days Storage

Adsorbents	Day1	Day2	Day3
Silica Gel	ND	ND	ND
	ND	ND	ND
	ND	ND	ND
Activated Carbon	ND	ND	ND
	ND	ND	ND
	ND	ND	ND
Sodium Bisulfite	236720	212330	184300
	260540	213050	222000
	240690	193230	162760
Cellulose	221080	199190	166110
	193250	158010	126240
	236730	203660	163820
Lactose	231660	203590	171210
	249200	221300	153480
	257300	207530	152830
Pectin	258560	223470	185080
	248070	207300	185430
	245310	197470	181260

*All samples were three replications.

Table C-4. Area Response of Hexanal in Cooperation with Adsorbents after 3 Days Storage

Adsorbents	Day1	Day2	Day3
Silica Gel	334560	129670	23241
	258170	127320	41274
	243030	76010	59992
Activated Carbon	260320	49952	13933
	220140	42357	43394
	204560	81885	61438
Sodium Bisulfite	242810	128680	10197
	472240	247590	36238
	325310	150149	53568
Cellulose	374220	218840	138350
	238640	152410	102380
	293540	143240	105000
Lactose	320920	188650	102180
	341320	231860	168270
	229550	229300	169280
Pectin	446730	234360	173750
	392230	331710	283070
	411400	333080	163250

*All samples were three replications.

Table C-5. Relative % Concentration of Butanal in Cooperation with Adsorbents after 3 Days Storage.

Adsorbents	Day1	Day2	Day3
Silica Gel	0.7689	0.1832	0.0000
	0.1455	0.0000	0.0000
	4.3208	1.7969	0.4546
Activated	0.1650	0.0000	0.0000
Carbon	0.9163	0.5691	0.2857
	0.0982	0.0000	0.0000
Sodium	34.9548	22.8643	10.9828
Bisulfite	14.4154	1.0594	0.1574
	39.5422	39.5928	21.5120
Cellulose	66.9969	53.8169	47.1420
	76.3321	61.2154	51.8898
	88.1382	77.6916	62.2935
Lactose	86.1968	76.0029	63.5051
	91.0074	81.2048	71.9101
	88.1382	77.6916	62.2935
Pectin	99.9205	91.8112	74.7197
	92.6937	94.4205	84.0979
	99.2527	86.2826	70.5697

*All samples were three replications.

Table C-6. Relative % Concentration of Cycloheptatriene in Cooperation with Adsorbents after 3 Days Storage

Adsorbents	Day1	Day2	Day3
Silica Gel	0.3333	0.1986	0.2432
	0.1699	0.1481	0.0000
	0.1852	0.1243	0.0000
Activated	0	0	0
Carbon	0	0	0
	0	0	0
Sodium	97.6517	91.3025	89.8341
Bisulfite	99.7243	69.4172	80.9542
	98.3004	82.4598	69.8655
Cellulose	91.2579	88.6430	82.4920
	94.3508	81.3021	70.8361
	83.1085	73.9612	66.2650
Lactose	94.7173	95.5047	85.8176
	99.9670	84.8939	76.7445
	98.7685	95.3339	76.8906
Pectin	92.5307	83.6484	78.9509
	99.2142	86.9121	82.3260
	93.6277	808700	68.7635

*All samples were three replications.

Table C-7. Relative % Concentration of Pentanal in Cooperation with Adsorbents after 3 Days Storage

Adsorbents	Day1	Day2	Day3
Silica Gel	0	0	0
	0	0	0
	0	0	0
Activated	0	0	0
Carbon	0	0	0
	0	0	0
Sodium	90.6175	81.2809	70.5509
Bisulfite	99.7359	81.5565	84.2170
	92.1372	73.9693	62.3052
Cellulose	84.6304	76.2508	63.5876
	73.9770	60.4869	48.3252
	90.6213	77.9619	62.7110
Lactose	88.6805	77.9352	65.5399
	95.3949	84.7146	58.7528
	96.5816	79.4434	58.5040
Pectin	98.9779	85.5453	60.9322
	94.9623	79.3554	70.9834
	93.9058	75.5924	69.3871

*All samples were three replications.

Table C-8. Relative % Concentration of Hexanal in Cooperation with Adsorbents after 3 Days Storage

Adsorbents	Day1	Day2	Day3
Silica Gel	69.7974	27.0523	4.8486
	53.8606	26.5621	8.6108
	50.7020	15.8575	12.5158
Activated	54.3091	10.4212	2.9068
Carbon	45.9266	8.8367	9.0530
	42.6762	17.0832	12.8175
Sodium	50.6561	26.8458	2.1273
Bisulfite	98.5208	51.6500	7.5601
	67.8676	31.3229	11.1756
Cellulose	78.0715	45.6554	28.8632
	49.7862	31.7965	21.3950
	61.2396	29.8834	21.9056
Lactose	66.9918	39.3570	21.3172
	71.2077	48.3717	35.1052
	47.8898	47.8376	35,3160
Pectin	93.1988	48.8932	36.2485
	81.8288	69.2028	59.0553
	85.8281	69.4887	34.0580

*All samples were three replications.

Table C-9. Area Response of Volatile Standards in the Headspace after 24, 48, and 72 Hours Storage at 21°C

Adsorbent	Time (hrs)	Butanal	Cyclohepta-triene	Pentanal	Hexanal
	24	3658,33	926.67	ND*	278586.67
Silica gel	48	1383.67	634.00	ND	111000.00
	72	317.67	327.33	ND	41502.33
	24	824.33	ND	ND	228340.00
Activated	48	397.67	ND	ND	58064.67
carbon	72	199.67	ND	ND	39588.33
	24	62131.00	398013.33	245983.33	346786.67
Sodium	48	44370.67	327346.67	206203.33	175473.00
Bisulfite	72	22817.00	323946.67	189020.00	33334.33
	24	161746.67	361723.33	217020.00	302133.33
Cellulose	48	134673.33	328325.00	186953.33	171496.67
	72	112732.33	295596.67	152056.67	115243.33
	24	190785.00	395020.00	244386.67	297263.33
Lactose	48	170235.00	371166.67	210806.67	216603.33
	72	150750.00	322330.00	159173.33	146576.67
	24	203953.33	384143.33	250646.67	416786.67
Pectin	48	190430.00	338453.33	209413.33	299716.67
	72	160293.33	309660.00	183923.33	206690.00

*ND: Not Detectable

APPENDIX D

APPENDIX D

Statistical Analysis

The statistical analysis was carried out by MSTATC microcomputer statistical program (Michigan State University, 1989).

Function: FACTOR

Experiment Model Number 5:

Completely Randomized Design for Factor A and B, Factor C is a Split Plot on A and B

Data case no. 1 to 216

Factorial ANOVA for the factors:

Replication (Var 1: replicate) with values from 1 to 3

Factor A (Var 2: volatile standard) with values from 1 to 4

Factor B (Var 3: adsorbent) with values from 1 to 6

Factor C (Var 4: day) with values from 1 to 3

Variable 5: GC

Grand Mean= 48.066 Grand Sum= 10382.202 Total Count= 216

TABLE OF MEANS

1	2	3	4	5	Total
*	1	*	*	42.712	2306.437
*	2	*	*	57.197	3088.613
*	3	*	*	51.961	2805.904
*	4	*	*	40.393	2181.248
*	*	1	*	7.746	278.844
*	*	2	*	5.724	206.065
*	*	3	*	56.908	2048.693
*	*	4	*	64.462	2320.638
*	*	5	*	73.886	2659.906
*	*	6	*	79.668	2868.056

*	1	1	*	0.852	7.670
*	1	2	*	0.226	2.034
*	1	3	*	20.565	185.081
*	1	4	*	65.057	585.516
*	1	5	*	81.374	732.367
*	1	6	*	88.187	793.769
*	2	1	*	0.156	1.403
*	2	2	*	0.000	0.000
*	2	3	*	86.612	779.512
*	2	4	*	81.357	732.216
*	2	5	*	89.849	808.638
*	2	6	*	85.205	766.843
*	3	1	*	0.000	0.000
*	3	2	*	0.000	0.000
*	3	3	*	81.819	736.370
*	3	4	*	70.483	634.345
*	3	5	*	78.394	705.547
*	3	6	*	81.071	729.642
*	4	1	*	29.975	269.771
*	4	2	*	22.670	204.030
*	4	3	*	38.637	347.730
*	4	4	*	40.951	368.560
*	4	5	*	45.928	413.354
*	4	6	*	64.200	577.802
*	*	*	1	59.807	4306.132
*	*	*	2	46.801	3369.671
*	*	*	3	37.589	2706.399
*	1	*	1	49.538	891.684
*	1	*	2	43.051	774.918
*	1	*	3	35.546	639.835
*	2	*	1	63.550	1143.907
*	2	*	2	56.365	1014.574
*	2	*	3	51.674	930.131
*	3	*	1	61.123	1100.222
*	3	*	2	51.894	934.093
*	3	*	3	42.866	771.589
*	4	*	1	65.018	1170.319
*	4	*	2	35.894	646.085
*	4	*	3	20.629	364.844

*	*	1	1	15.024	180.284
*	*	1	2	5.978	71.739
*	*	1	3	2.235	26.821
*	*	2	1	12.008	144.091
*	*	2	2	3.076	36.910
*	*	2	3	2.089	25.063
*	*	3	1	73.677	884.124
*	*	3	2	54.444	653.327
*	*	3	3	42.604	511.242
*	*	4	1	78.209	938.510
*	*	4	2	63.222	758.665
*	*	4	3	51.955	623.463
*	*	5	1	86.098	1033.181
*	*	5	2	74.751	897.006
*	*	5	3	60.810	729.718
*	*	6	1	93.828	1125.941
*	*	6	2	79.335	952.023
*	*	6	3	65.841	790.092
*	1	1	1	1.745	5.235
*	1	1	2	0.660	1.980
*	1	1	3	0.152	0.455
*	1	2	1	0.393	1.180
*	1	2	2	0.190	0.569
*	1	2	3	0.095	0.286
*	1	3	1	29.637	88.912
*	1	3	2	21.172	63.516
*	1	3	3	10.884	32.652
*	1	4	1	77.156	231.467
*	1	4	2	64.241	192.724
*	1	4	3	53.775	161.325
*	1	5	1	91.007	273.022
*	1	5	2	81.205	243.614
*	1	5	3	71.910	215.730
*	1	6	1	97.289	291.867
*	1	6	2	90.838	272.515
*	1	6	3	76.462	229.387
*	2	1	1	0.229	0.688
*	2	1	2	0.108	0.323
*	2	1	3	0.130	0.391
*	2	2	1	0.000	0.000
*	2	2	2	0.000	0.000
*	2	2	3	0.000	0.000

*	2	3	1	98.559	295.676
*	2	3	2	81.061	243.182
*	2	3	3	80.218	240.654
*	2	4	1	89.572	268.717
*	2	4	2	81.302	243.906
*	2	4	3	73.198	219.593
*	2	5	1	97.818	293.453
*	2	5	2	91.911	275.732
*	2	5	3	79.818	239.453
*	2	6	1	95.124	285.373
*	2	6	2	83.810	251.431
*	2	6	3	76.680	230.040
*	3	1	1	0.000	0.000
*	3	1	2	0.000	0.000
*	3	1	3	0.000	0.000
*	3	2	1	0.000	0.000
*	3	2	2	0.000	0.000
*	3	2	3	0.000	0.000
*	3	3	1	94.164	282.491
*	3	3	2	78.936	236.807
*	3	3	3	72.358	217.073
*	3	4	1	83.076	249.229
*	3	4	2	71.567	214.700
*	3	4	3	56.806	170.417
*	3	5	1	93.552	280.657
*	3	5	2	80.698	242.093
*	3	5	3	60.932	182.797
*	3	6	1	95.949	287.846
*	3	6	2	80.164	240.493
*	3	6	3	67.101	201.303
*	4	1	1	58.120	174.360
*	4	1	2	23.145	69.436
*	4	1	3	8.658	25.975
*	4	2	1	47.637	142.912
*	4	2	2	12.114	36.341
*	4	2	3	8.259	24.777
*	4	3	1	72.348	217.044
*	4	3	2	36.607	109.822
*	4	3	3	6.954	20.863
*	4	4	1	63.032	189.097
*	4	4	2	35.778	107.335
*	4	4	3	24.043	72.128
*	4	5	1	62.016	186.049

*	4	5	2	45.189	135.586
*	4	5	3	30.579	91.738
*	4	6	1	86.952	260.856
*	4	6	2	62.528	187.585
*	4	6	3	43.121	129.362

ANALYSIS OF VARIANCE TABLE

K Value	Source	Degrees of Freedom	Sum of Squares	Mean Square	F Value
2	Factor A	3	10048.009	3349.336	30.4868
4	Factor B	5	195515.242	39103.048	355.9291
6	AB	15	48740.349	3249.357	29.5767
-7	Error	48	5273.371	109.862	
8	Factor C	2	17944.602	8972.301	285.9067
10	AC	6	6677.277	1112.880	35.4624
12	BC	10	2427.733	242.773	7.7361
14	ABC	30	2188.714	72.957	2.3248
-15	Error	96	3012.664	31.382	
	Total	215	291827.962		

Conditional Comparisons

The judgment of significance depends on the difference between any two means. Any value of the difference between two means larger than minimum significant difference (MSD) will be considered significant.

There are two MSD; one for the volatile standards and the other for the adsorbents. The following equations are employed to calculate MSD.

$$t = (\bar{y}_1 - \bar{y}_2) / \sqrt{2\hat{\sigma}^2 / 3}$$

$$MSD = \left(t_{B,0.025,m=6,\hat{\upsilon}}\right)\sqrt{2\hat{\sigma}^2 / 3}$$

Equations for volatile standards

$$t = (\bar{y}_1 - \bar{y}_2) / \sqrt{2\hat{\sigma}^2 / 3}$$

$$MSD = \left(t_{B,0.025,m=15,\hat{\upsilon}}\right)\sqrt{2\hat{\sigma}^2 / 3}$$

Equations for adsorbents

$$\hat{\sigma}^2 = (MS_{E1} + 2MS_{E2})/3$$

$$\hat{\upsilon} = [\hat{\sigma}^2]^2 / \left\{ \frac{(MS_{E1}/3)^2}{48} + \frac{(2MS_{E2}/3)^2}{96} \right\}$$

$\hat{\sigma}^2$ = [109.862 + 2(31.382)]/3 = 57.542

$\hat{\upsilon}$ = $(57,542)^2$ / { [$(109.862/3)^2$ /48] + [$(2x31.382/3)^2$ /96] }
= 101.886

$t_{B,0.025,m=6,\hat{\upsilon}} = t_{B,0.025,m=6,101.886}$ = 2.692 (Gill, 1987)

$t_{B,0.025,m=15,\hat{\upsilon}} = t_{B,0.025,m=15,101.886}$ = 3.007 (Gill, 1987)

MSD for volatile standards = $(2.692)\sqrt{\frac{2(57.542)}{3}}$ = 16.674

MSD for adsorbents = $(3.007)\sqrt{\frac{2(57.542)}{3}}$ = 18.625

Within the same day (three days) and the same adsorbent (six adsorbents), comparison between any two out of four volatile standards was executed as the following. The difference between two means ($\overline{y_1} - \overline{y_2}$) corresponding to the volatile standards was compared to MSD for significant difference.

<u>Day1</u>

Within Silica Gel	$\overline{y_1} - \overline{y_2}$
Butanal vs. Cycloheptatriene	1.745 - 0.229 = 1.516
Butanal vs. Pentanal	1.745 - 0.000 = 1.745
Butanal vs. Hexanal	1,745 - 58.120 = 56.375
Cycloheptatriene vs. Pentanal	0.229 - 0.000 = 0.229
Cycloheptatriene vs. Hexanal	0.229 - 58.120 = 57.891
Pentanal vs. Hexanal	0.000 - 58.120 = 58.120

Within Activated Carbon	$\bar{y}_1 - \bar{y}_2$
Butanal vs. Cycloheptatriene	0.393 - 0.000 = 0.393
Butanal vs. Pentanal	0.393 - 0.000 = 0.393
Butanal vs. Hexanal	0.393 - 47.637 = 47.244
Cycloheptatriene vs. Pentanal	0.000 - 0.000 = 0.000
Cycloheptatriene vs. Hexanal	0.000 - 47.637 = 47.637
Pentanal vs. Hexanal	0.000 - 47.637 = 47.637

Within Sodium Bisulfite	$\bar{y}_1 - \bar{y}_2$
Butanal vs. Cycloheptatriene	29.637 - 98.559 = 68.922
Butanal vs. Pentanal	29.637 - 94.164 = 64.527
Butanal vs. Hexanal	29.637 - 72.348 = 42.711
Cycloheptatriene vs. Pentanal	98.559 - 94.164 = 4.395
Cycloheptatriene vs. Hexanal	98.559 - 72.348 = 26.211
Pentanal vs. Hexanal	94.164 - 72.348 = 21.816

Within Cellulose	$\bar{y}_1 - \bar{y}_2$
Butanal vs. Cycloheptatriene	77.156 - 89.572 = 12.416
Butanal vs. Pentanal	77.156 - 83.076 = 5.92
Butanal vs. Hexanal	77.156 - 63.032 = 14.124
Cycloheptatriene vs. Pentanal	89.572 - 83.076 = 6.496
Cycloheptatriene vs. Hexanal	89.572 - 63.032 = 26.54
Pentanal vs. Hexanal	83.076 - 63.032 = 20.044

Within Lactose	$\bar{y}_1 - \bar{y}_2$
Butanal vs. Cycloheptatriene	91.007 - 97.818 = 6.811
Butanal vs. Pentanal	91.007 - 93.552 = 2.545
Butanal vs. Hexanal	91.007 - 62.016 = 28.991
Cycloheptatriene vs. Pentanal	97.818 - 93.552 = 4.266
Cycloheptatriene vs. Hexanal	97.818 - 62.016 = 35.802
Pentanal vs. Hexanal	93.552 - 62.016 = 31.536

Within Pectin	$\bar{y}_1 - \bar{y}_2$
Butanal vs. Cycloheptatriene	97.289 - 95.124 = 2.165
Butanal vs. Pentanal	97.289 - 95.949 = 1.340
Butanal vs. Hexanal	97.289 - 86.952 = 10.337
Cycloheptatriene vs. Pentanal	95.124 - 95.949 = 0.825
Cycloheptatriene vs. Hexanal	95.124 - 86.952 = 8.172
Pentanal vs. Hexanal	95.949 - 86.952 = 8.997

Day2

Within Silica Gel	$\overline{y_1} - \overline{y_2}$
Butanal vs. Cycloheptatriene	0.660 - 0.108 = 0.552
Butanal vs. Pentanal	0.660 - 0.000 = 0.660
Butanal vs. Hexanal	0.660 - 23.145 = 22.485
Cycloheptatriene vs. Pentanal	0.108 - 0.000 = 0.108
Cycloheptatriene vs. Hexanal	0.108 - 23.145 = 23.037
Pentanal vs. Hexanal	0.000 - 23.145 = 23.145

Within Activated Carbon	$\overline{y_1} - \overline{y_2}$
Butanal vs. Cycloheptatriene	0.190 - 0.000 = 0.190
Butanal vs. Pentanal	0.190 - 0.000 = 0.190
Butanal vs. Hexanal	0.190 - 12.144 = 11.954
Cycloheptatriene vs. Pentanal	0.000 - 0.000 = 0.000
Cycloheptatriene vs. Hexanal	0.000 - 12.144 = 12.144
Pentanal vs. Hexanal	0.000 - 12.144 = 12.144

Within Sodium Bisulfite	$\overline{y_1} - \overline{y_2}$
Butanal vs. Cycloheptatriene	21.172 - 81.061 = 59.889
Butanal vs. Pentanal	21.172 - 78.936 = 57.764
Butanal vs. Hexanal	21.172 - 36.607 = 15.435
Cycloheptatriene vs. Pentanal	81.061 - 78.936 = 2.125
Cycloheptatriene vs. Hexanal	81.061 - 36.607 = 44.454
Pentanal vs. Hexanal	78.936 - 36.607 = 42.329

Within Cellulose	$\overline{y_1} - \overline{y_2}$
Butanal vs. Cycloheptatriene	64.241 - 81.302 = 17.061
Butanal vs. Pentanal	64.241 - 71.567 = 7.326
Butanal vs. Hexanal	64.241 - 35.778 = 28.463
Cycloheptatriene vs. Pentanal	81.302 - 71.567 = 9.735
Cycloheptatriene vs. Hexanal	81.302 - 35.778 = 45.524
Pentanal vs. Hexanal	71.567 - 35.778 = 35.789

Within Lactose	$\overline{y_1} - \overline{y_2}$
Butanal vs. Cycloheptatriene	81.205 - 91.911 = 10.706
Butanal vs. Pentanal	81.205 - 80.698 = 0.507
Butanal vs. Hexanal	81.205 - 45.189 = 36.016
Cycloheptatriene vs. Pentanal	91.911 - 80.698 = 11.213
Cycloheptatriene vs. Hexanal	91.911 - 45.189 = 46.722
Pentanal vs. Hexanal	80.698 - 45.189 = 35.509

Within Pectin	$\bar{y}_1 - \bar{y}_2$
Butanal vs. Cycloheptatriene	90.838 - 83.810 = 7.028
Butanal vs. Pentanal	90.838 - 80.164 = 10.674
Butanal vs. Hexanal	90.838 - 62.528 = 28.310
Cycloheptatriene vs. Pentanal	83.810 - 80.164 = 3.646
Cycloheptatriene vs. Hexanal	83.810 - 62.528 = 21.282
Pentanal vs. Hexanal	80.164 - 62.528 = 17.636

Day3

Within Silica Gel	$\bar{y}_1 - \bar{y}_2$
Butanal vs. Cycloheptatriene	0.152 - 0.130 = 0.022
Butanal vs. Pentanal	0.152 - 0.000 = 0.152
Butanal vs. Hexanal	0.152 - 8.658 = 8. 506
Cycloheptatriene vs. Pentanal	0.130 - 0.000 = 0.130
Cycloheptatriene vs. Hexanal	0.130 - 8.658 = 8.528
Pentanal vs. Hexanal	0.000 - 8.658 = 8.658

Within Activated Carbon	$\bar{y}_1 - \bar{y}_2$
Butanal vs. Cycloheptatriene	0.095 - 0.000 = 0.095
Butanal vs. Pentanal	0.095 - 0.000 = 0.095
Butanal vs. Hexanal	0.095 - 8.259 = 8.164
Cycloheptatriene vs. Pentanal	0.000 - 0.000 = 0.000
Cycloheptatriene vs. Hexanal	0.000 - 8.259 = 8.259
Pentanal vs. Hexanal	0.000 - 8.259 = 8.259

Within Sodium Bisulfite	$\bar{y}_1 - \bar{y}_2$
Butanal vs. Cycloheptatriene	10.884 - 80.218 = 69.334
Butanal vs. Pentanal	10.884 - 72.358 = 61.474
Butanal vs. Hexanal	10.884 - 6.954 = 3.930
Cycloheptatriene vs. Pentanal	80.218 - 72.358 = 7.860
Cycloheptatriene vs. Hexanal	80.218 - 6.954 = 73.264
Pentanal vs. Hexanal	72.358 - 6.954 = 65.404

Within Cellulose	$\bar{y}_1 - \bar{y}_2$
Butanal vs. Cycloheptatriene	53.775 - 73.198 = 19.423
Butanal vs. Pentanal	53.775 - 56.806 = 3.031
Butanal vs. Hexanal	53.775 - 24.043 = 29.732
Cycloheptatriene vs. Pentanal	73.198 - 56.806 = 16.392
Cycloheptatriene vs. Hexanal	73.198 - 24.043 = 49.155
Pentanal vs. Hexanal	56.806 - 24.043 = 32.763

Within Lactose	$\overline{y_1} - \overline{y_2}$
Butanal vs. Cycloheptatriene	71.910 - 79.818 = 7.908
Butanal vs. Pentanal	71.910 - 60.932 = 10.978
Butanal vs. Hexanal	71.910 - 30.579 = 41.331
Cycloheptatriene vs. Pentanal	79.818 - 60.932 = 18.886
Cycloheptatriene vs. Hexanal	79.818 - 30.579 = 49.239
Pentanal vs. Hexanal	60.932 - 30.579 = 30.353

Within Pectin	$\overline{y_1} - \overline{y_2}$
Butanal vs. Cycloheptatriene	76.462 - 76.680 = 0.218
Butanal vs. Pentanal	76.462 - 67.101 = 9.361
Butanal vs. Hexanal	76.462 - 43.121 = 33.341
Cycloheptatriene vs. Pentanal	76.680 - 67.101 = 9.579
Cycloheptatriene vs. Hexanal	76.680 - 43.121 = 33.559
Pentanal vs. Hexanal	67.101 - 43.121 = 23.980

Within the same day (three days) and the same volatile standard (four volatile standards), comparison between any two out of six adsorbents was executed as the following. The difference between two means ($\overline{y_1} - \overline{y_2}$) corresponding to the adsorbents was compared to MSD for significant difference.

<u>Day1</u>

Within Butanal	$\overline{y_1} - \overline{y_2}$
Silica gel vs. Activated Carbon	1.745 - 0.393 = 1.352
Silica gel vs. Sodium Bisulfite	1.745 - 29.637 = 27.892
Silica gel vs. Cellulose	1.745 - 77.156 = 75.411
Silica gel vs. Lactose	1.745 - 91.007 = 89.262
Silica gel vs. Pectin	1.745 - 97.289 = 95.544
Activated Carbon vs. Sodium Bisulfite	0.393 - 29.637 = 29.244
Activated Carbon vs. Cellulose	0.393 - 77.156 = 76.763
Activated Carbon vs. Lactose	0.393 - 91.007 = 90.614
Activated Carbon vs. Pectin	0.393 - 97.289 = 96.896
Sodium Bisulfite vs. Cellulose	29.637 - 77.156 = 47.519
Sodium Bisulfite vs. Lactose	29.637 - 91.007 = 61.370
Sodium Bisulfite vs. Pectin	29 637 - 97.289 = 67.652
Cellulose vs. Lactose	77.156 - 91.007 = 13.851
Cellulose vs. Pectin	77.156 - 97.289 = 20.133
Lactose vs. Pectin	91.007 - 97.289 = 6.282

Within Cycloheptatriene	$\overline{y_1} - \overline{y_2}$
Silica gel vs. Activated Carbon	0.229 - 0.000 = 0.229
Silica gel vs. Sodium Bisulfite	0.229 - 98.559 = 98.330
Silica gel vs. Cellulose	0.229 - 89.572 = 89.343
Silica gel vs. Lactose	0.229 - 97.818 = 97.589
Silica gel vs. Pectin	0.229 - 95.124 = 94.895
Activated Carbon vs. Sodium Bisulfite	0.000 - 98.559 = 98.559
Activated Carbon vs. Cellulose	0.000 - 89.572 = 89.572
Activated Carbon vs. Lactose	0.000 - 97.818 = 97.818
Activated Carbon vs. Pectin	0.000 - 95.124 = 95.124
Sodium Bisulfite vs. Cellulose	98,559 - 89.572 = 8.987
Sodium Bisulfite vs. Lactose	98.559 - 97.818 = 0.741
Sodium Bisulfite vs. Pectin	98.559 - 95.124 = 3.435
Cellulose vs. Lactose	89.572 - 97.818 = 8.246
Cellulose vs. Pectin	89.572 - 95.124 = 5.552
Lactose vs. Pectin	97.818 - 95.124 = 2.694

Within Pentanal	$\overline{y_1} - \overline{y_2}$
Silica gel vs. Activated Carbon	0.000 - 0.000 = 0.000
Silica gel vs. Sodium Bisulfite	0.000 - 94.164 = 94.164
Silica gel vs. Cellulose	0.000 - 83.076 = 83.076
Silica gel vs. Lactose	0.000 - 93.552 = 93.552
Silica gel vs. Pectin	0.000 - 95.949 = 95.949
Activated Carbon vs. Sodium Bisulfite	0.000 - 94.164 = 94.164
Activated Carbon vs. Cellulose	0.000 - 83.076 = 83.076
Activated Carbon vs. Lactose	0.000 - 93.552 = 93.552
Activated Carbon vs. Pectin	0.000 - 95.949 = 95.949
Sodium Bisulfite vs. Cellulose	96.164 - 83.076 = 13.088
Sodium Bisulfite vs. Lactose	96.164 - 93.552 = 2.612
Sodium Bisulfite vs. Pectin	96.164 - 95.949 = 0.215
Cellulose vs. Lactose	83.076 - 93.952 = 10.476
Cellulose vs. Pectin	83.076 - 95.949 = 12.873
Lactose vs. Pectin	93.552 - 95.949 = 2.397

Within Hexanal	$\overline{y_1} - \overline{y_2}$
Silica gel vs. Activated Carbon	58.120 - 47.637 = 10.483
Silica gel vs. Sodium Bisulfite	58.120 - 72.348 = 14.228
Silica gel vs. Cellulose	58.120 - 63.032 = 4.912
Silica gel vs. Lactose	58.120 - 62.016 = 3.896
Silica gel vs. Pectin	58.120 - 86.952 = 28.832
Activated Carbon vs. Sodium Bisulfite	47.637 - 72.348 = 24.711

Activated Carbon vs. Cellulose	47.637 - 63.032 = 15.395
Activated Carbon vs. Lactose	47.637 - 62.016 = 14.379
Activated Carbon vs. Pectin	47.637 - 86.952 = 39.315
Sodium Bisulfite vs. Cellulose	72.348 - 63.032 = 9.316
Sodium Bisulfite vs. Lactose	72.348 - 62.016 = 10.332
Sodium Bisulfite vs. Pectin	72.348 - 86.952 = 14.604
Cellulose vs. Lactose	63.032 - 62.016 = 1.204
Cellulose vs. Pectin	63.032 - 86.952 = 23.920
Lactose vs. Pectin	63.032 - 86.952 = 24.936

Day2

Within Butanal

	$\bar{y}_1 - \bar{y}_2$
Silica gel vs. Activated Carbon	0.660 - 0.190 = 0.470
Silica gel vs. Sodium Bisulfite	0.660 - 21.172 = 20.512
Silica gel vs. Cellulose	0.660 - 64.241 = 63.581
Silica gel vs. Lactose	0.660 - 81.205 = 80.545
Silica gel vs. Pectin	0.660 - 90.838 = 90.178
Activated Carbon vs. Sodium Bisulfite	0.190 - 21.172 = 20.982
Activated Carbon vs. Cellulose	0.190 - 64.241 = 64.051
Activated Carbon vs. Lactose	0.190 - 81.205 = 81.015
Activated Carbon vs. Pectin	0.190 - 90.838 = 90.648
Sodium Bisulfite vs. Cellulose	21.172 - 64.241 = 43.069
Sodium Bisulfite vs. Lactose	21.172 - 81.205 = 60.033
Sodium Bisulfite vs. Pectin	21.172 - 90.838 = 69.666
Cellulose vs. Lactose	64.241 - 81.205 = 16.964
Cellulose vs. Pectin	64.241 - 90.838 = 26.597
Lactose vs. Pectin	81.205 - 90.838 = 9.633

Within Cycloheptatriene

	$\bar{y}_1 - \bar{y}_2$
Silica gel vs. Activated Carbon	0.108 - 0.000 = 0.108
Silica gel vs. Sodium Bisulfite	0.108 - 81.061 = 80.953
Silica gel vs. Cellulose	0.108 - 81.302 = 81.194
Silica gel vs. Lactose	0.108 - 91.911 = 91.803
Silica gel vs. Pectin	0.108 - 83.810 = 83.702
Activated Carbon vs. Sodium Bisulfite	0.000 - 81.061 = 81.061
Activated Carbon vs. Cellulose	0.000 - 81.302 = 81.302
Activated Carbon vs. Lactose	0.000 - 91.911 = 91.911
Activated Carbon vs. Pectin	0.000 - 83.810 = 83.810
Sodium Bisulfite vs. Cellulose	81.061 - 81.302 = 0.241
Sodium Bisulfite vs. Lactose	81.061 - 91.911 = 10.850
Sodium Bisulfite vs. Pectin	81.061 - 83.810 = 2.749
Cellulose vs. Lactose	81.302 - 91.911 = 10.609

Cellulose vs. Pectin	81.302 - 83.810 = 2.508
Lactose vs. Pectin	91.911 - 83.810 = 8.101

Within Pentanal

	$\overline{y}_1 - \overline{y}_2$
Silica gel vs. Activated Carbon	0.000 - 0.000 = 0.000
Silica gel vs. Sodium Bisulfite	0.000 - 78.936 = 78.936
Silica gel vs. Cellulose	0.000 - 71.567 = 71.567
Silica gel vs. Lactose	0.000 - 80.698 = 80.698
Silica gel vs. Pectin	0.000 - 80.164 = 80.164
Activated Carbon vs. Sodium Bisulfite	0.000 - 78.936 = 78.963
Activated Carbon vs. Cellulose	0.000 - 71.567 = 71.567
Activated Carbon vs. Lactose	0.000 - 80.698 = 80.698
Activated Carbon vs. Pectin	0.000 - 80.164 = 80.164
Sodium Bisulfite vs. Cellulose	78.936 - 71.567 = 7.369
Sodium Bisulfite vs. Lactose	78.936 - 80.698 = 1.762
Sodium Bisulfite vs. Pectin	78.936 - 80.164 = 1.228
Cellulose vs. Lactose	71.567 - 80.698 = 9.131
Cellulose vs. Pectin	71.567 - 80.164 = 8.597
Lactose vs. Pectin	80.698 - 80.164 = 0.534

Within Hexanal

	$\overline{y}_1 - \overline{y}_2$
Silica gel vs. Activated Carbon	23.145 - 12.114 = 11.031
Silica gel vs. Sodium Bisulfite	23.145 - 36.607 = 13.462
Silica gel vs. Cellulose	23.145 - 35.778 = 12.633
Silica gel vs. Lactose	23.145 - 45.189 = 22.044
Silica gel vs. Pectin	23.145 - 62.528 = 39.383
Activated Carbon vs. Sodium Bisulfite	12.114 - 36.607 = 24.493
Activated Carbon vs. Cellulose	12.114 - 35.778 = 23.664
Activated Carbon vs. Lactose	12.114 - 45.189 = 33.075
Activated Carbon vs. Pectin	12.114 - 62.528 = 50.414
Sodium Bisulfite vs. Cellulose	36.607 - 35.778 = 0.289
Sodium Bisulfite vs. Lactose	36.607 - 45.189 = 8.582
Sodium Bisulfite vs. Pectin	36.607 - 62.528 = 25.921
Cellulose vs. Lactose	35.778 - 45.189 = 9.411
Cellulose vs. Pectin	35.778 - 62.528 = 26.750
Lactose vs. Pectin	45.189 - 62.528 = 17.339

<u>Day3</u>

Within Butanal

	$\overline{y}_1 - \overline{y}_2$
Silica gel vs. Activated Carbon	0.152 - 0.095 = 0.057

Silica gel vs. Sodium Bisulfite	0.152 - 10.884 = 10.732
Silica gel vs. Cellulose	0.152 - 53.775 = 53.623
Silica gel vs. Lactose	0.152 - 71.910 = 71.758
Silica gel vs. Pectin	0.152 - 76.462 = 76.310
Activated Carbon vs. Sodium Bisulfite	0.095 - 10.884 = 10.789
Activated Carbon vs. Cellulose	0.095 - 53.775 = 53.680
Activated Carbon vs. Lactose	0.095 - 71.910 = 71.815
Activated Carbon vs. Pectin	0.095 - 76.462 = 76.367
Sodium Bisulfite vs. Cellulose	10.884 - 53.775 = 42.891
Sodium Bisulfite vs. Lactose	10.884 - 71.910 = 61.026
Sodium Bisulfite vs. Pectin	10.884 - 76.462 = 65.578
Cellulose vs. Lactose	53.775 - 71.910 = 18.135
Cellulose vs. Pectin	53.775 - 76.462 = 22.687
Lactose vs. Pectin	71.910 - 76.462 = 4.552

Within Cycloheptatriene	$\overline{y}_1 - \overline{y}_2$
Silica gel vs. Activated Carbon	0.130 - 0.000 = 0.130
Silica gel vs. Sodium Bisulfite	0.130 - 80.210 = 80.080
Silica gel vs. Cellulose	0.130 - 73.198 = 73.068
Silica gel vs. Lactose	0.130 - 79.818 = 79.688
Silica gel vs. Pectin	0.130 - 76.680 = 76.550
Activated Carbon vs. Sodium Bisulfite	0.000 - 80.210 = 80.210
Activated Carbon vs. Cellulose	0.000 - 73.198 = 73.198
Activated Carbon vs. Lactose	0.000 - 79.818 = 79.818
Activated Carbon vs. Pectin	0.000 - 76.680 = 76.680
Sodium Bisulfite vs. Cellulose	80.210 - 73.198 = 7.012
Sodium Bisulfite vs. Lactose	80.210 - 79.818 = 0.392
Sodium Bisulfite vs. Pectin	80.210 - 76.680 = 3.530
Cellulose vs. Lactose	73.198 - 79.818 = 6.620
Cellulose vs. Pectin	73.198 - 76.680 = 3.482
Lactose vs. Pectin	79.818 - 76.680 = 3.318

Within Pentanal	$\overline{y}_1 - \overline{y}_2$
Silica gel vs. Activated Carbon	0.000 - 0.000 = 0.000
Silica gel vs. Sodium Bisulfite	0.000 - 72.358 = 72.358
Silica gel vs. Cellulose	0.000 - 56.806 = 56.806
Silica gel vs. Lactose	0.000 - 60.932 = 60.932
Silica gel vs. Pectin	0.000 - 67.101 = 67.101
Activated Carbon vs. Sodium Bisulfite	0.000 - 72.358 = 72.358
Activated Carbon vs. Cellulose	0.000 - 56.806 = 56.806
Activated Carbon vs. Lactose	0.000 - 60.932 = 60.932
Activated Carbon vs. Pectin	0.000 - 67.101 = 67.101

Sodium Bisulfite vs. Cellulose	72.358 - 56.806 = 15.552
Sodium Bisulfite vs. Lactose	72.358 - 60.932 = 11.426
Sodium Bisulfite vs. Pectin	72.358 - 67.101 = 5.257
Cellulose vs. Lactose	56.806 - 60.932 = 4.126
Cellulose vs. Pectin	56.806 - 67.101 = 10.295
Lactose vs. Pectin	60.932 - 67.101 = 6.169

Within Hexanal	$\overline{y}_1 - \overline{y}_2$
Silica gel vs. Activated Carbon	8.658 - 0.393 = 0.399
Silica gel vs. Sodium Bisulfite	8.658 - 6.954 = 1.704
Silica gel vs. Cellulose	8.658 - 24.043 = 15.385
Silica gel vs. Lactose	8.658 - 30.579 = 21.921
Silica gel vs. Pectin	8.658 - 43.121 = 34.463
Activated Carbon vs. Sodium Bisulfite	8.259 - 6.954 = 1.305
Activated Carbon vs. Cellulose	8.259 - 24.043 = 15.784
Activated Carbon vs. Lactose	8.259 - 30.579 = 22.320
Activated Carbon vs. Pectin	8.259 - 43.121 = 34.862
Sodium Bisulfite vs. Cellulose	6.954 - 24.043 = 17.089
Sodium Bisulfite vs. Lactose	6.954 - 30.579 = 23.625
Sodium Bisulfite vs. Pectin	6.954 - 43.121 = 36.167
Cellulose vs. Lactose	24.043 - 30.579 = 6.536
Cellulose vs. Pectin	24.043 - 43.121 = 19.078
Lactose vs. Pectin	30.579 - 43.121 = 12.542

APPENDIX E

APPENDIX E

CONSENT FORM

SCHOOL OF PACKAGING

MICHIGAN STATE UNIVERSITY

PRODUCT INGREDIENTS: FROZEN FISH (LAKE TROUT), SILICA GEL, ACTIVATED CARBON

I,_____, have read the above list or ingredients and find none that I know I am allergic to. I have also been informed of the nature of the research, including experimental materials and procedures, which will be used during the sensory testing sessions. I agree to serve on this sensory panel, which is being conducted on this _____ day of _____, 1993. In addition to sniffing samples, I will be asked to complete a brief questionnaire. I understand that I am free to withdraw my consent and to discontinue participation in the panel at any time without penalty. I understand that if I am injured as a result of my participation in this research project, Michigan State University will provide emergency medical care, if necessary, but these and any other medical expenses must be paid from my own health insurance program.

SIGNED _____ DATE _____

QUESTIONNAIRE FOR SIMPLE PAIRED COMPARISONS TEST

NAME: _____ DATE: _____

PRODUCT: FROZEN FISH (LAKE TROUT)

Evaluate the off-odor of these two samples of frozen lake trout.

Please follow the instructions stated below:

a. When ready to begin, remove the cap carefully from the first bottle on your left.

b. Without delay, inhale deeply from the open top of the sample bottle using nostrils only (mouth should be closed).

c. After sniffing, replace the seal quickly.

d. Take several deep breaths before going on to the next sample.

e. Repeat steps a. through c. for the other sample.

f. Indicate which sample has stronger off-odor.

g. If no difference is apparent, enter your best guess.

Test Pairs	Which sample has stronger off-odor

Comments: _____

_____

_____

_____

_____

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