# Chemical and Physical Properties of "İsot" Type Pepper During Production

**University of Gaziantep** 

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**M.Sc.Thesis** 

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## ABSTRACT

## CHEMICAL AND PHYSICAL PROPERTIES OF "ISOT" TYPE PEPPER DURING PRODUCTION

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"Isot" is a kind of spice which is produced from red pepper and has a special color (black, red, and purple), smell, and taste. It is used in various products. İsot production can be achieved in two ways; industrial and traditional methods. In this study, the analyses and relationships of color, acidity, total phenolic content, ascorbic acid, antioxidant content, pH, non-enzymatic browning, and capsaicin content of isot pepper obtained from industrially simulated at 25,35,45°C,open atmosphere conditions; traditionally simulated and factory produced samples were examined from fresh pepper samples to final "isot" product by time.

All samples generally showed an decrease in pH and increase in the acidity, decrease in moisture content, increase in the ASTA (American Spice Trade Association) color values, fluctuations in L\*, a\*, b\* values, decrease in ascorbic acid content, increase in non-enzymatic browning index, decrease in antioxidant content, fluctuations in total phenolic content, and increase in the capsaicin by time.

Key words: "İsot" pepper, color, non-enzymatic browning index, capsaicin.

## ÖZET

## İSOT TİPİ BİBER ÜRETİMİ SIRASINDAKİ BAZI FİZİKSEL VE KİMYASAL ÖZELLİKLER

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İsot kırmızıbiberden elde edilen ve kendine has renk (siyah - kırmızı-mor), tat, koku ve aroması olan bir baharattır.Bir çok üründe kullanılmaktadır. İsot üretimi endüstriyel ve geleneksel olmak üzere ikiye ayrılır.Bu çalışmadaendüstriyel ve geleneksel yöntemler kullanılarak değişik sıcaklıklarda (25,35,45°C ve açık havada) üretilmiş isot biberinin ve fabrikadan alınmış orijinal numunelerin ham maddeden son ürün oluşumuna kadarki süreçte alınan numunelerinde nem,renk, asitlik, toplam fenolik madde, askorbik asit,antioksidant, pH,enzimatik olmayan kararma,ve "capcaicin" tayinleri yapılarak bu süreçteki değişimler gözlenmiş ve birbirleriyle olan ilişkileri incelenmiştir.

İsot oluşum aşamasında pH değerinde bir azalma ve asitlik değerinde bir artma, in L\*, a\*, b\* değerlerinde dalgalanmalarASTArenk değerinde bir artma, toplam fenolik madde miktarlaında dalgalanmalar, capsaicin miktarında bir artma, C vitamini değerinde bir azalma, antioksidan miktarında birazalma ve enzimatik olmayan kararma indeksinde bir artış görülmektedir.

Anahtar Kelimeler: ISOT biberi, renk, enzimatik olmayan kararma ve "capsaicin"

TO MY FATHER and MOTHER

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## ABREVIATIONS

| AOAC  | Association of Official Analytical Chemists |
|-------|---|
| ASTA  | American Spice Trade Association            |
| ANOVA | Analysis of Variance                        |
| a*    | Redness-greenness                           |
| b*    | Yellowness-darkness                         |
| L*    | Lightness-darkness                          |
| FAO   | Food and Agricultural Organization          |
| С     | Capsaicin                                   |
| DHC   | Dihydrocapcaicin                            |
| DPPH  | 2,2-diphenyl-1-picrylhydracyl               |
| Ι     | Industrial                                  |
| HDHC  | Homodihydrocapcaicin                        |
| НС    | Homocapcaicin                               |
| NDHC  | Nordihydrocapcaicin                         |
| PAVA  | Nonivamide                                  |
| ST    | Simulated Traditional                       |
| SI    | Simulated Industrial                        |

### **CHAPTER I**

#### **INTRODUCTION**

"Isot" is a traditional spice and widely used particularly in southeastern part of Turkey. For a long time, "isot" was produced traditionally for family consumption and partly marketing. Nowadays modern technology has increased the product quality and improved storage conditions. So isot becomes popular.

"İsot" is a kind of hot spices which has a special taste, aroma and color, produced from *Capsicum annuum L*. Production of "İsot" is discussed in section 2.2 in details.

Red peppers have been consumed for 2000 years as fresh, dried, pepper flakes, chili powders and recent years "isot". The differences between isot and other pepper products are isot's characteristics taste and aroma.

Red pepper which is used for "isot" production comes from Solanaceae family and botanic name is *Capsicum annuum L.*, is grown in many places in the world. Chilli and paprika belong to *capsicum annuum* species, while the peppers which are very hot belong to *capsicum frutescens* species. Capsicum is the common name of *Capsicum annuum* and *Capsicum frutescens* species (Govindarajan, 1985).

In Turkey, hot red pepper production for spices changes according to years between 20-40 million tons. In terms of production amounts, first one is pepper flakes, the second one is powder type and the third one is the isot production (8-12 % of the total production). This portion is usually consumed in "lahmacun" and particularly in "çiğköfte".

The red pepper for isot making is usually grown in Kahramanmaraş, Gaziantep, Şanlıurfa and Hatay. The red peppers are nearly 35-40 g weight and 8-12 cm height, 5-6 cm diameter. Fruits initially have dark green color and then they turn into red color when they are reached maturation.

Drying is the main processing step of 1sot production. Drying technique is also important for isot production which affects the quality and properties of end product.

Drying can either be done by traditional sun drying or industrially through the use of solar dryers or hot air drying systems.

There is little information found in literature about isot. ((Poyrazoğlu et al., 2005, (Erdoğan, 2004)) However, there are a lot of information about red pepper spices. Most of the studies are related to drying process. İsot and dried red pepper could show some similarities in physical and chemical properties because of the similar raw material and process.

## 1.1.Aim of the research

In this work isot was prepared in laboratory conditions similar to industrial production at 25 °C, 35 °C, 45 °C and industrial and traditional simulations at open atmosphere condition. The moisture content, pH, acidity, color, non enzymatic browning, vitamin C content, total phenolic content, capsaicinoid content, antioxidant content changes were observed every day during the production period.

Therefore the aim of the research was to;

1. Simulate the isot production at laboratory conditions

2. Monitor the following parameters; moisture content, pH, acidity, color, extractable color, non enzymatic browning, vitamin C content, total phenolic content, capsaicinoid content, antioxidant content during isot production.

3. Compare the results with industrially produced samples.

## **CHAPTER II**

## LITERATURE REVIEW

## 2.1. Red Pepper(Paprika (Capsicum annuum L.)

Capsicum is a genus of flowering plants in the nightshade family Solanaceae. Its species are native to the Americas where they have been cultivated for thousands of years. In modern times, it is cultivated worldwide, and is mainly used in many regional cuisines. In addition to use as spices and food vegetables, capsicum has also found a use in medicines.

The fruit of Capsicum plants have a variety of names depending on place and type. The spicy variety is commonly called chili peppers, or simply "chilies". The large mild form is called red pepper, green pepper or bell pepper in North America, sweet pepper in Britain, and typically just "capsicum" in Australia, New Zealand, and India. The fruit is called paprika in some other countries (although paprika can also refer to the powdered spice made from various capsicum fruit).

Assumed that today there are nearly 20 types of pepper. The most knowns are *Capsicum annuum, Capsicum frutescens, Capsicum chinense, Capsicum pendulum and Capsicum pubescens.* Shapes and colors of different varieties of peppers are shown in Figure 2.1.



Figure 2.1. Peppers in different shapes and colors belongs to Capsicum

## 2.1.1. Pepper Composition

The chemical composition of peppers for raw green, raw red and chilli peppers is shown in Table 2.1. The species had a significant effect on the chemical composition. For example, energy content of peppers ranged from 21 to 113 Kcals.

|                   | Raw green | Raw red | Chilies |
|-------------------|-----------|---------|---------|
| Water (g)         | 92.1      | 92.1    | 92.5    |
| Energy (kcal)     | 113       | 113     | 21      |
| Fat (g)           | 0.19      | 0.19    | 0.10    |
| Protein(g)        | 0.89      | 0.89    | 0.90    |
| Carbohydrates (g) | 6.43      | 6.43    | 5.3     |
| Fiber (g)         | 1.8       | 2       | 1.3     |
| Potassium (mg)    | 177       | 177     | 187     |
| Phosphorous (mg)  | 19        | 19      | 17      |
| Magnessium (mg)   | 10        | 10      | 14      |
| Calcium (mg)      | 9         | 9       | 7       |
| Vitamin C (mg)    | 89.3      | 190     | 68      |
| Vitamin B2 (mg)   | 0.03      | 0.03    | 0.05    |
| Vitamin B6 (mg)   | 0.248     | 0.248   | 0.153   |
| Vitamin A (IU)    | 632       | 5700    | 610     |
| Vitamin E (mg)    | 0.69      | 0.69    | 0.69    |
| Niacine (mg)      | 0.5       | 0.5     | 0.8     |

## Table2.1.Composition of pepper for 100 gr

Source: USDA Nutrient Data Base

The hot pepper has a number of i ndustrial uses as can be seen in Table 2.2.

| INDUSTRY (end user)                | PRODUCT TYPE                | PEPPER<br>PRODUCTS<br>USED |  |
|------------------------------------|-----------------------------|----------------------------|--|
| Spice and Condiment industry       | Pepper in Brine             | Fresh pepper               |  |
|                                    | Sauces & salsas             | Ground pepper              |  |
|                                    | Seasonings                  | Hot pepper<br>mash         |  |
| Ethnic Food Industry (mainly in    | Processed meats             | Grounded                   |  |
| European market)                   | Hot sauces                  | pepper                     |  |
|                                    | Dips                        | Hot pepper<br>mash         |  |
|                                    | Candy                       |                            |  |
| Pharmaceutical Industry            | Topical creams              | Capsaicin oil, oleoresins  |  |
| Health & Wellness Industry         | Capsules                    | Grounded pepper            |  |
| Defence industry                   | Stun guns                   | Capsaicin oil,             |  |
|                                    | Pepper sprays               | oleoresins                 |  |
| Fiery Foods Industry (mainly in US | Extra hot sauces ( not for  | Capsaicin oil,             |  |
| market)                            | numan consumption )         | oleoresins                 |  |
| Restaurant and Food Catering       | Gourmet<br>sauces Marinades | Fresh pepper               |  |
| industry                           | Table sauces                | Hot pepper<br>mash         |  |
|                                    |                             | Grounded hot<br>pepper     |  |
| Home Décor Industry                | Decorative pieces           | Fresh pepper               |  |

Source::http://www.unctad.info/en/Infocomm/AACP-Products/COMMODITY-PROFILE---Spices/

## 2.1.2. Importance of Pepper In The World Market

The top nine pepper producing countries are listed in Figure 2.2 below. (FAO, 2009).China is the top producers of Chillies & Pepper and . Turkey is in the third line in isot production after China and Mexica in the World(Figure 2.2.).



Figure 2.2. Production of pepper in the World (FAO, 2009).

Production of "Pepper" has not been relatively stable over the years in Turkey. In 2010, production stood at 186000 tones (TÜİK, 2011). The production areas and amounts are listed in Table 2.3.

| Year | Area(decare) | Production | Year | Area(decare) | Production |
|------|--------------|------------|------|--------------|------------|
|      |              | (Ton)      |      |              | (Ton)      |
| 1988 | 63 670       | 20 953     | 2000 | 80 940       | 21 340     |
| 1989 | 86 200       | 21 303     | 2001 | 90 000       | 20 000     |
| 1990 | 83 610       | 24 216     | 2002 | 70 000       | 30 000     |
| 1991 | 68 020       | 20 921     | 2003 | 100 000      | 40 000     |
| 1992 | 60 790       | 16 136     | 2004 | 71 600       | 30 000     |
| 1993 | 56 400       | 14 492     | 2005 | 78 000       | 45 000     |
| 1994 | 58 810       | 14 878     | 2006 | 66 960       | 45 861     |
| 1995 | 57 840       | 13 887     | 2007 | 71 285       | 67 213     |
| 1996 | 61 520       | 14 750     | 2008 | 77 747       | 60 000     |
| 1997 | 63 200       | 14 466     | 2009 | 91 372       | 196 900    |
| 1998 | 61 900       | 14 333     | 2010 | 104 049      | 186 272    |
| 1999 | 57 730       | 16 080     |      |              |            |

Table 2.3. Production area of red pepper and production amount

Source:(2011 TÜİK Spice Report)

## 2.1.3. Kahramanmaraş type red pepper

Kahramanmaraş region (including Adıyaman, Gaziantep, Kilis, Şanlıurfa) is major production area of red hot pepper (Figure 2.3) as spices and isot pepper. Because of its own specific color, aroma, taste, and pungency, high quality spices are produced.



Figure 2.3.Kahramanmaraş type red pepper

## 2.2. Technology of isot production

There are two kinds of production of isot. One of them is traditional production the other one is the industrial production.



### Figure 2.4. Flow chart of traditional isot production

Production scheme of traditional isot is given in Figure 2.4. Firstly raw red peppers which are suitable for isot production are cleaned and their stems and seeds are removed after that they are spread out onto concrete for one day to withering and discoloration. They are then put in to PVC bags or covered with the PVC bags are opened during the day time and closed during the night time (Figure 2.5). This process continues until the peppers are dried. The drying process takes 5 to 9 days depending on the weather conditions. After drying peppers are grinded and mixed with salt and vegetable oil. The desired isot (bright and well-colored) is then obtained.



Figure 2.5. Photos of traditional isot production.

(Taken from http://www.kemalvuraltarlan.com/isot-story.html)

Production scheme of industrial isot is given in Figure 2.6. There are some differences between traditional and industrial isot production. Firstly raw red peppers (having 82-87 % moisture content) which are suitable for isot production are taken and their stems are separated but seeds are not removed. They cut in to 1 cm thickness. If the isot production will be done later, moisture content is decreased to 4% at 54-74°C by a hot air drying systems. If it is processed immediately, step-5 is skipped. The dried red peppers and dried seeds are tempered until they reach to 20-30% moisture content. They are then grinded according to desired size are mixed (30 % seed+ 70 % dried fruit). The mixture is passed through screw to compress them. Aim of this part is to increase the temperature. After compressing, the mixture of pepper fruit and seeds are put in to the bags covered with tent canvases. This prevents heat losses. In this step, temperature is increased to nearly 94 °C step by itself and the temperature starts to decrease. Desired isot is obtained when moisture content is reached to 12 %.

Fresh red pepper (82-87% moisture content) – step 1 Cleaning - step 2 Sorting stems - step 3 Cutting into 1 cm thickness - step 4 Drying (until 4% moisture content, at 54-74°C) - step 5 Tempering dried red peppers and dried seeds until reach 20-30% moisture contentstep 6 Grinding of pepper seeds and fruits - step 7 Mixing (30 % seed+ 70 % dried fruit) - step 8 Compress in helix with screws - step 9 (Temperature rise nearly 54 °C) Mixture is put in to the bags over and over and covered with canvas - step 10 (Temperature is increase to nearly 94 °C step by step and after this point temperature is again start to decrease and moisture content reached to 12 %) T Addition of salt (6%) and oil (6%) - step 11

Packaging - step 12

Figure 2.6. Flow chart of industrial isot production

Figure 2.7 shows industrial isot production. Washing, cleaning and drying processes are shown only.



Figure 2.7. Photo of Industrial İsot Production

## 2.3. Characteristic of red pepper

## 2.3.1. Pungency

Pungency is a strong odor or taste property referred to as hotness for chilli pepper.

Pungency is based on the concentration of capsaicinoids, primarily of capsaicin. The capsaicin is concentrated in the placenta area (white pith where seeds are attached). 60% of capsaicin is in the white pith and 40% in the seeds.

Capsaicin is the main capsaicinoid in chili peppers, followed by dihydrocapsaicin. These two compounds are also about twice as potent to the taste and nerves as the minor capsaicinoids, nordihydrocapsaicin, homodihydrocapsaicin and homo capsaicin. Table 2.4 shows properties of various capcaicinoids. Pure capsaicin is a hydrophobic, colorless, odorless, and crystalline to waxy compound.

| Capcaicinoid name    | Abbrev. | Typical  | Scoville   | Chemical  |
|----------------------|---------|----------|------------|---|
|                      |         | relative | heat units | structure   |
|                      |         | amount   |            |   |
| Capsaicin            | С       | 69%      | 16,000,000 | HO HO HI HO |
| Dihydrocapsaicin     | DHC     | 22%      | 16,000,000 | HO H H H H H H H H H H H H H H H H H H  |
| Nordihydrocapsaicin  | NDHC    | 7%       | 9,100,000  | HO HO HI HI HI HI HI HI HI HI HI HI HI HI HI  |
| Homodihydrocapsaicin | HDHC    | 1%       | 8,600,000  | HO H H H H H H H H H H H H H H H H H H  |
| Homocapsaicin        | HC      | 1%       | 8,600,000  | HO LO H   |
| Nonivamide           | PAVA    |          | 9,200,000  | HO J J J J J J J J J J J J J J J J J J J  |

## Table 2.4. Properties of capcaicinoids

## 2.3.2. Color

The intense and characteristic red color of Capsicum fruits is principally due to the pigments of capsanthin and capsorubin. They belong to carotenoids, which are present in the chloroplast of red pepper as partially esterified with fatty acids.

Capsanthin and capsorubin are the major red pigments and are unique to the capsicum species. Capsanthin, which is commonly esterified with fatty acids as monoesters and diesters, as well as in free form, may represent as much as 50% of the total carotenoids (Locey and Guzinski, 2000). Especially capsanthin is regarded as a functional material because of its properties of stimulating singlet oxygenquenching ability (Hirayama et al., 1994) and preventing colon carcinogenesis.

# 2.4. Changes in the characteristics of 1sot during production 2.4.1. Drying

Dehydration is an important preservation process which reduces water activity through the decrease of water content, avoiding potential deterioration and contamination during long storage periods. Also, food quality of is preserved, the hygienic conditions are improved, and product loss is diminished (Kaymak and Ertekin, 2002).

A very common method of preservation for these agricultural corps is to dry them in order to conserve the perishable fruits, reduce storage volume and to extend their shelf life beyond the few weeks when they are in season Drying can either be done by traditional sun drying or industrially through the use of solar dryers or hot air drying (Riva and Peri, 1986). However both traditional and industrial sun-drying methods (i.e. drying with forms of solar energy) though having low operational installation and energy costs, still are labor intensive because of climatic variations. The use of solar drying may result in quality degradation of the final dehydrated products. The use of hot-air drying is a viable option for drying fruits and vegetables because of the large amounts of fruits produced annually which have to be dried for preservation and the fact that climatic factors do not affect the drying process.

Several factors can influence hot-air drying of food, for example: velocity and temperature of air, water diffusion through material, load density, thickness and shape of the product to be dried. However, the sudden removal of water decreases the nutritional and sensorial value of food, and allows the presence of phenomena such as hardening and shrinkage and it is well known that during hot-air drying, vegetables undergo physical, structural, chemical and nutritional changes that can affect quality attributes like texture, color, flavor, and nutritional value (Di Scala and Crapiste, 2008).

The increasing demand for high-quality shelf-stable dried vegetables requires the design, simulation and further optimization of the drying process with the purpose of accomplishing not only the efficiency of the process but also the final quality of the dry product. During drying, vegetables undergo physical, structural, chemical, organoleptic and nutritional changes that cause quality degradation (Crapiste, 2000; Di Scala and Crapiste, 2008). An adequate preservation method must decrease the

water activity (up to values where microbial activity and other reactions are inhibited) and maintain the texture, mechanical properties, nutritional values, physical properties, etc.

Some studies cited in the literature have addressed the influence of drying conditions on the quality characteristics of the dehydrated product (Simal et al., 2005). Conventional drying of pepper causes a major loss of color and texture quality of the final product. Undesirable changes in the color may lead to a decrease in its quality and marketing value, therefore, the surface color of the pepper is an important criterion. Loss of red color is caused by autoxidation of carotenoids. The stability of the main carotenoids of the red bell pepper during storage has been shown to depend on the drying conditions, with the rate of deterioration increasing as the drying temperature increases (Doymaz and Pala, 2002; Vega GaLvez et al., 2008).

Kim et al.(2006) dried red peppers at 80 °C for 5h, then 60 °C for 18 h or 70 °C for 6h; short time and low temperature drying of cutpods, was reported to be more effective than conventional drying in reducing the destruction of the antioxidant activity, ascorbic acid and color.

Scala and Crapiste (2008) dried a thin layer of red pepper samples in across flow laboratory scale dryer with air at 50, 60 and 70 °C, ambient relative humidity and 0.2 and 1.2 m/s air velocity. They represented the drying kinetics by a diffusive model, the effective moisture diffusivity ranging from 5.01 to  $8.32 \times 10^{-10}$  m<sup>2</sup>/s with activation energy of 23.35 kJ/mol.

Tunde and Akintunde et al. (2005) dried red bell-peppers using sun, solar and artificial (i.e. hot-air) drying. The drying curves and rates obtained indicated that drying of bell-pepper was done in two drying rate periods, the constant drying rate period (mainly) and the falling drying rate period. The existence of the constant drying rate period was more pronounced in the artificial air-drying method than in the other two drying methods and the drying process was also faster. This was attributed to the effect of temperature of drying air on the diffusion of water from internal regions to the surface of the product.

Arslan and Özcan (2010) were dried red bell peppers with oven, sun and microwave oven drying and observed change in drying behavior, color and antioxidant content.

The sun, oven (50 and 70 °C) and microwave oven (210 and 700W) drying behaviors of red bell-pepper slices were investigated. Effects of these drying methods in terms of color indices and antioxidant activity of pepper slices were also studied. Sun dried and followed by microwave oven (700W) dried samples revealed the highest L\*, a\* and b\* color values than the other dried samples. Microwave oven dried (210W) and oven dried (50 °C) samples exhibited the lowest Trolox equivalent antioxidant capacity (TEAC) and DPPH radical scavenging activities among the dried samples.

Doymaz and Pala (2002) was studied drying kinetics of red peppers under different pretreatment and air drying conditions. Red peppers, *Capsicum annuum L*, grown in the region of Kahramanmaras, Turkey, were pretreated with various solutions of alkaline emulsion of ethyl oleate (AEEO) and dried afterwards. Results indicated that pretreated peppers dried faster and had higher Hunter L\*(lightness), a\* (redness) and b\* (yellowness) values than the untreated and dried peppers. 2% ethyl oleate and 5% K<sub>2</sub>CO<sub>3</sub> solution was found to be the most effective dipping solution that provided for whole peppers dried at 50 °C, and yielded best color quality.

#### 2.4.2. Moisture content

Pepper is categorized as perishable food due to its high moisture content but by drying it will be more stable, durable and nonperishable. It is known that isot production is based on drying process. Drying is defined as a process of moisture removal due to simultaneous heat and mass transfer .The initial moisture content of raw red pepper which is suitable for isot production is between 82-87%. The final moisture content of isot is around 12%. İsot production involves initial drying process in which moisture content of fresh pepper is decreased from 82-87% to 20-30%. Isot is produced from peppers of 20-30% of moisture content.

#### 2.4.3. Texture

The acceptability of dried products depends mainly on their structural properties (such as e texture), which is one of the attributes used by consumers in judging their quality. Destruction of the cellular system during drying causes important physical and structural changes.

#### 2.4.4. Color

*Capsicum annuum L* show a tremendous variation in fruit and foliage size, shape, and color, and there are hundreds of varieties (Bailes, 1999). Pepper varieties differ greatly in pungency and color due to various breeding environments such as cultivating area, heat levels, water amounts and temperature. Furthermore, the deterioration of red pepper during processing is also a serious problem, affected by many factors including variety, moisture content, ripeness stage, and healthy state of dry fruits before grinding and drying condition. These affect the color of final products such as pepper powder.

Carotenoids pigments give their color to pepper. Drying process decreases the stability of the carotenoid pigments (Biacs et al., 1992).

Kim et al. (2004) studied the composition of main carotenoids in Korean red pepper (*Capsicum annuum*, *L*.) and changes of pigment stability during the drying and storage process This study was conducted to identify main carotenoids in red pepper and to investigate pigment stability during drying and storage. The main pigments in saponified extracts were capsanthin, zeaxanthin,  $\beta$ -cryptoxanthin,  $\beta$ -carotene, and an unknown peak, which was considered to be capsorubin by spectrophotometry and liquid chromatography/mass spectrometry (LC/MS). Myristoylcapsanthin, lauroylmyristoy lcapsanthin, and myristoylpalmitoylcapsanthin were identified from unsaponified extracts analyzing fragment ions by LC/MS.

Vega-Galvez et al. (2009) studied the effects of process temperatures between 50 and 90°C on physico-chemical properties, rehydration, colour, texture, vitamin C, antioxidant capacity and total phenolics during the drying of red pepper. The rehydration ratio decreased with temperature and the maximum water holding capacity was achieved at 50 °C. Both vitamin C content and the total phenolic content decreased as air-drying temperature decreased. The radical scavenging activity showed higher antioxidant activity at high temperatures (i.e. 80 and 90°C) rather than at low temperatures (i.e. 50, 60 and 70°C). Chromatic parameters (L\*, a\*, b\*, C\* and H), non-enzymatic browning compounds and extractable color were affected by drying temperature, which contributed to the discoloring of pepper during this process.

#### **2.4.5. Time and temperature**

Vega et al., (2009) studied the drying kinetics of the red bell pepper (var. Lamuyo) and modeled at different temperatures (50, 60, 70 and 80°C), using an air velocity of 2.5 m s<sup>-1</sup>. Only the falling rate period was observed during the experiment. Effective moisture diffusivity was estimated to be between  $3.2 \times 10^{-9}$  and  $11.2 \times 10^{-9}$  m<sup>2</sup> s<sup>-1</sup>within the temperature range studied. The effect of temperature on the diffusivity was described by the Arrhenius equation with activation energy (Ea) of 39.70 kJ mol<sup>-1</sup>. The Newton, Henderson–Pabis, Page and Page modified models were applied to the experimentally obtained moisture data. Comparisons of the experimental values with the calculated values demonstrated that the Page modified model produced the best fit for every drying curve, representing an excellent tool for estimation of the drying time.

Vega Galvez et al. (2008) dried red peppers at four air inlet temperatures from 50 to 80  $\circ$ C and rehydrated in water at 30  $\circ$ C to study the influence of air drying temperature on the quality and micro structural properties of the rehydrated tissue and also determined effects of sample pretreatment (immersing in a solution containing NaCl<sub>2</sub>CaCl<sub>2</sub> and Na<sub>2</sub>S<sub>2</sub>O<sub>5</sub> prior to drying) and suggested that damage to cellular structure was minimized by pretreatment of samples; the resulting rehydrated peppers displayed comparatively improved vitamin C retention, color and firmness.

### 2.4.6. Capsaicinoid content

Capsaicinoids present in red peppers cause pungent, hot-tasting, sensations when consumed as part of the diet. The concentration of capsaicinoids in hot pepper varieties ranges from 0.003 to 0.01%; varieties of mild chillies contain from 0.5 to 0.3%, and strong chillies are characterized by a content higher than 0.3%, reaching about 1%, Perucka and Oleszek (2000).

Kurian and Starks (2002) are studied analysis of capsaicin and dihydrocapsaicin in whole chili peppers by HPLC. Peppers, both undried and dried, contained average values of 1250 ppm capsaicin and 540 ppm dihydrocapsaicin relative to undried weight. Relative to dry weight, the orange habañero peppers contained 8840 ppm capsaicin and 3940 ppm dihydrocapsaicin.

Topuz and Özdemir (2007) were studied assessment of carotenoids, capsaicinoids and ascorbic acid composition of some selected pepper cultivars (*Capsicum annuum* 

*L.*) grown in Turkey by means of HPLC technique. The findings determined that the cultivars of 730 F1 and 1245 F1 had higher carotenoids (2310–2390 mg/kg in dry basis), capsaicinoids (471.3–688.1 mg/kg in dry basis), vitamin A (218.8–243.0 mg RAE/100 g in wet basis) and vitamin C (63.1–64.9 mg/100 g in wet basis) content, without any significant difference among each of them. Furthermore, the cultivars which had higher capsaicinoids contents had higher ascorbic acids content as well.

## **CHAPTER III**

## MATERIALS AND METHODS

## 3.1. Materials

Main material fresh Kahramanmaraş type (*Capsicum annuum L*) red pepper was bought from wholesale market of Gaziantep. After washing, cleaning and sorting processes, the drying process was conducted.

İsot samples produced by an industrial method were taken from Ölmez Agricultural Products Factory (Gaziantep, Turkey) every day during production.

All of the samples which are taken day by day are analyzed in the same day if not they were stored at -40°C until they were analyzed.

All chemicals used in this study were of analytical grade and purchased from Merck Chemical Co. (Darmstadt, Germany)

The samples named "**Industrial**, (**I**)" means the products obtained through the way as discussed in Figure 2.6 and discussed in details at section 2.2. This is a real industry production.

In this study, the sample named "**Simulated Traditional, (ST**)" means the products obtained through traditional way as discussed in Figure 2.4 and details are given at section 2.2.

In addition to these two methods, industrial production way was simulated at laboratory conditions with 3 different temperatures (25°C, 35°C, 45°C) and under atmospheric conditions. These are indicated as "Simulated Industrial, (SI)". "Kepertme" part in industrial production was also tried to be simulated by packing the samples tightly and pressurized by putting a stone on top of packages. For example, samples processed at 25 °C, 35°C, and 45°C are shown as "SI-25""SI-35""SI-45", respectively.

#### **3.2. Methods**

#### **3.2.1.** Moisture content determination

The moisture content was determined by the oven method according to Official Method of Analysis (AOAC, 1995). The measurements were carried out in triplicate.

## 3.2.2. Extractable color determination

The ASTA color value was measured using the ASTA Analytical Method 20.1 (1986). The ASTA color of the red pepper samples was measured by a spectrophotometer (Perkin Elmer Lampda 25Model). Red pepper powder (70–100 mg) was added to 100 mL of acetone, and then the mixture was stored at 0 °C for 4 h with intermittent stirring. The absorbance of an aliquot of the transparent extract was measured at 460 nm using a UV/VIS spectrophotometer. ASTA color value was calculated using the following equation:

$$ASTA color value = \frac{absorbance of acetone extracts * 16.4 * If}{sample weight(g)}$$

Where "**If**" is a correction factor for the apparatus, which was calculated from the absorbance of a standard solution of potassium dichromate, ammonium sulphate and cobalt sulphate.

#### **3.2.3. Surface color determination**

The surface color measurement of isot samples were performed using Hunter Lab Color Flex (A60-1010-615 Model Colorimeter, Hunter Lab, and Reston VA) during production. The L\*, a\*, b\* color values (also referred to as CIELAB) were used to express the color changes. The color values are express as L\* (darkness/whiteness), a\* (greenness/redness), b\* (blueness/yellowness) values. Three measurements were taken for all samples and average values were shown. The instrument was calibrated against the standard reference white tile "L\*=93.41, a\*=-1.12, b\*=1.07".

## **3.2.4.DPPH radical scavenging activity determination**

Antioxidant activity was evaluated by measuring the radical scavenging effect of dried peppers methanolic extracts towards the2,2-diphenyl-1-picrylhydracyl (DPPH)

as reported previously by Singh et al. (2002) and Bamdad et al. (2006). 5mL of a 0.1mM methanol solution of DPPH (Fluka) was added to 0.1mL of methanol extracts of pepper samples. The tubes were allowed to stand at 27 °C for 20min. The decrease in absorbance at 517nm was recorded in a spectrophotometer (Perkin Elmer Lampda 25Model).Radical scavenging activity was expressed as inhibition percentage and was calculated using the following formula:

percent radical scavening activity = 
$$\left(\frac{\text{control OD} - \text{sample OD}}{\text{control OD}}\right) \times 100$$

## **3.2.5. Total Phenolic Content Determination**

The total phenolic content of isot samples were determined by Folin-Ciocalteu Method (Singleton, Orthofer, Lamuela and Raventos, 1999) 250  $\mu$ l of Folin-Ciocalteu reagent was added to 50  $\mu$ l of sample. After 2 min. 750  $\mu$ l of 20% (w/v) aqueous Na<sub>2</sub>CO<sub>3</sub> solution was added and the volume was made up to 5.0 mL with water. The controls contained all the reaction reagents except the extract. After 2 h of incubation at 20 °C, absorbance was measured at 765 nm and compared with the gallic acid calibration curve. Total phenol content was determined as gallic acid equivelent mg gallic acid/g extract, and the value was presented as the mean of duplicate analysis.

#### **3.2.6.** Non Enzymatic Browning Index Determination

The methodology applied for determination of non-enzymatic browning compounds (NEB) solubilized in the rehydration water was proposed by Vega-Gálvez et al. (2008b). The rehydration water was first clarified by centrifugation at 3200×g for 10 min. The supernatant was diluted with an equal volume of ethanol at 95% and centrifuged again at 3200×g for 10 min. The browning index (absorbance at 420 nm) of the clear extracts was determined in quartz cuvettes using a spectrophotometer (Perkin Elmer Lampda 25 Model). All measurements were done in duplicate.

#### **3.2.7.** Vitamin C Determination

Vitamin C (Ascorbic Acid, AA) was determined based upon the quantitative discoloration of 2,6-dichlorophenol indophenols titrimetric method as described in AOAC methodology No. 967.21 (AOAC, 2000). The vitamin C content was
expressed as mg AA retained/100 g dry matter. All measurements were done in duplicate.

#### 3.2.8. pH determination

The pH value of the sample was determined using a pH meter (Jenway 3010; Jenway LTD, Essex, UK) equipped with a J95, 924001 electrode (Jenway LTD, Essex,UK). The measurement of pH value was carried out on 1 g of sample homogenized in distilled water (1/10: sample/water).

## **3.2.9.** Acidity Determination

Titratable acidity was determined by potentiometric titration against 0.1 N NaOH to pH 8.2 and expressed in malic acid equivalents, the predominant acid in ripe bell pepper (Lunning et al., 1994)

#### **3.2.10.** Capcaicin Determination

Capsaicinoids were extracted from the samples of red pepper pastes by applying the technique described by Collins et al., 1995. The capsaicinoids were extracted from 2 g sample in 12mL acetonitrile by heating at 80 °C for 4 h. Suspensions were periodically shaken every 30 min throughout the extraction process. The suspended material was allowed to cool and settle. The supernatant was filtered into a 2mL glass vial using 0.45 mm membrane filter (Millipore) and used for HPLC injections. Liquid chromatography was performed using a HPLC solvent delivery system equipped with a fluorescence detector (RF-20 A, JAPAN) and Star software for data processor. The separation was performed on a Nucleosil 5 C18 column (250×4.6mm i.d.) coupled with a Nucleosil 5 C18 guard column (4×4.6mm i.d.). The following HPLC operating condition, used by Peusch et al. (1997), was employed. The eluent was a mixture of acetonitrile/water/acetic acid (100:100:1) at a flow rate of 1.2 mL/min. The fluorescence detector was set at 280nm excitations and 320 nm emissions. Injection volumes, run time and temperature were 20 mL, 23 min and 20-22 °C, respectively. Standards of capsaicin (98%), dihydrocapsaicin (90%) and mixture of capsaicinoids (60% capsaicin) were used for retention time verification and quantification.

## **CHAPTER IV**

## **RESULT AND DISCUSSION**

## **4.1. Moisture Content**

Isot production is a kind of drying process. This provides desired texture, color, aroma and longer shelf life. The samples were dried with different methods and different temperatures discussed in section 3.1. The initial moisture content of our fresh pepper was 91.5% and it was decreased to nearly 13%. One way ANOVA analysis showed that (in appendix) a significant decrease in moisture content was observed(p<0,05).



Figure 4.1.Plot of moisture content versus production days

The moisture content of the material was very high during the initial phase of the drying which resulted in high drying rates. Afterwards, moisture content for all samples was almost constant. In this study, drying rate calculations were not done. That is why, it is hard to say anything about the length and presence of falling and constant rate periods. However, Arslan and Özcan, (2010) reported that the entire

drying process for the red pepper samples occurred in the range of falling rate period. Kaymak and Ertekin, (2002), Ade and Omowaye et al., (2003) reported that drying process took place in the falling rate period,

Drying times for all methods were selected as the same for all process (except original industrial sample). When the moisture contents are compared with respect to drying temperatures, small differences between dryings at 25, 35 and 45°C were observed but the moisture content decreases with the increasing temperature. When the drying methods are compared there are no significant differences (p>0.05) between simulated traditional and simulated industrial method according to statistical analyses.

### 4.2. Color of Isot

Pepper colors can be examined with the following ways (Tepić and Vujičić, 2004):1. surface (apparent) color, 2.extractable (ASTA) color, and 3. carotenoids profiles. Extractable and surface color measurements are the main quality methods used in the spice industry.

Type, degree of ripeness, granulation and processing methods are all affecting the final appearance of pepper. ASTA color affects the brightness of a product, while the surface color has an impact on the hue of the product. Hue sets the kind of color, e.g. brownish-red, orange-red cultivar.

Photos of pepper samples of simulated industrial production at 45 °C and those of industrial production are shown in Figure 4.2 and Figure 4.3, respectively. It is easy to follow formation of dark color at industrial production by time. However, the same dark color formation was not followed for simulated industrial products. Necessary conditions (lack of the followings: compact pressing during drying, use of large batches during processing and enough increase in temperature to approximately 90 °C during "kepertme") were not occurred for dark color formation.

Temperatures of the simulated industrial samples were constant at 25, 35, 45 °C throughout the isot production. However, in industrial production, temperatures of samples could have reached to 90-95 °C during "kepertme" process. In our simulation of industrial isot production, temperatures were lower than 90-95 °C, therefore expected biochemical reactions would have not been occurred.



**Figure 4.2.** Pictures of peppers during simulated industrial production at 45  $^{\circ}$ C



Figure 4.3 Pictures of peppers during industrial production (from factory production)

"1. Gün"=Day 1 shows the grinded samples time just after the mechanically drying with moisture contents of 20-25%.

"3. Gün"= Day 3 shows the samples after compressing in screw (temperature rise nearly 54 °C),

"5. Gün"=Day 5 shows the samples after packaging in bags and covering them with canvas (temperature rise nearly 94 °C),

"8. Gün"= Day 8 shows the samples after addition of salt (6%) and oil (6%) and packaging

## 4.2.1. Surface Color

Surface color information provides to specify colors perceived by the human eye. It is very difficult to describe colors verbally, because two people might describe the same color by different terms. The light source and illumination, the back ground color and contrast, and the angle at which the object is viewed will affect specification of the color. Surface color of the peppers varies according to type, growing conditions, dehydration, milling and storage conditions.

Surface color measurement was determined by Hunter Lab Colorimeter. The color brightness coordinate  $L^*$  measures the whiteness value of a color and ranges from black at 0 to white at 100. The chromaticity coordinate  $a^*$  measures red when positive and green when negative, and chromaticity coordinate  $b^*$  measures yellow when positive and blue when negative (Doymaz et al., 2006).



Figure 4.4.Plot of L values versus production days

Changes in L values of isot samples in production process by time are shown in Figure 4.4. Day 1 indicates the fresh samples. In general there are fluctuations in L-values of all samples. In all the conditions there was a decrease between the first day and the last day samples significantly (p<0.05 in appendix). All of the samples lost their brightness during isot production process. When the process was compared according to temperatures they were shown similar fluctuations. At the end of the processes, L\* values decrease from 32.2 to 17.7 for the original isot sample and to 9.79 for the sample which was processed at 45°C, respectively.

The decrease in  $L^*$  values can be attributed to brown pigment formation during drying as Park and Lee (1975) reported that brown pigment in dried red peppers was due to their high levels of reducing sugars and amino acids in red pepper.



Figure 4.5. Plot of a\* values versus production days

Changes in 'a' values of isot samples in production process by time are shown in Figure 4.5. In general there were fluctuations in a \* values for all type of samples significantly (p<0,05 in appendix), but a decrease in 'a' value from the first sample to the last sample was observed. It is a fact that isot samples lost their redness on their surfaces and turned to brownish color. The biggest change in a\* value from initial to final stage was observed from 32.51 to 3.24 for the industrial sample. Arslan and Özcan (2010) dried red bell peppers by different methods, Results indicated that a\* values of fresh and sun dried samples were higher than microwave and oven dried samples

Changes in b values of isot samples in production process are shown in Figure 4.6. In general there were fluctuations in all type of sample significantly (p<0,05 in appendix) but a decrease in a value of b\* from the first sample to last sample was observed. The maximum change between the initial and final stages was observed at the ones processed at 45° C of simulated industrial samples and the minimum change was in simulated traditional samples. In particular, the b\* values suddenly decreased at day 4 (from 26.71 to 3.33) due to the decrease of redness caused by rapid discoloring during sunlight exposure for traditionally processed samples, After 4 days b\* value increased to 10.05 at the end of isot production.



Figure 4.6. Plot of b\* values versus production days

## 4.2.2. Extractable Color

Extractable color is a measure of total pigment content. Current procedures for measuring extractable color (total pigments) in dehydrated capsicums and oleoresins were developed and approved by the Association of Official Analytical Chemists (AOAC) and the American Spice Trade Association (ASTA). Extractable color is measured using a spectrophotometer and is expressed in ASTA units. Generally, the higher the ASTA color value, the greater the effect on the brightness or richness of the final product.

The present study showed that the ASTA values significantly (P < 0.05 in appendix) changed by drying methods. Change in ASTA color values of isot samples during production by traditional and industrial methods and 25°C, 35°C, 45°C are shown in Figure 4.7.

All samples were shown a similar increase trend. The highest ASTA value was recorded in pepper processed simulated industrially at 45 °C with the values of 189.5. In contrast, the, industrial and simulated industrial methods for the pepper yielded the lowest ASTA values without significant differences between each other. In industrial simulation process with different temperatures, there is an increase in ASTA color values in increasing temperature.



Figure 4.7. Plot of ASTA color value versus production days

The increase in ASTA values could be attributed to biosynthesis of carotenoids in living tissues. This phenomenon was also reported in previous research for slow drying processes (Minguez and Mosquera and Hornero-Mendez, 1994 and Minguez and Mosquera et al., 1994). The ASTA values of industrial samples were lower than those for all drying methods. The industrial isot production at the factory was being produced by oven drying. This provided better drying conditions.

Loss of red color is caused by autoxidation of carotenoids. The stability of the main carotenoids of the red bell pepper during storage has been shown to depend on the drying conditions, with the rate of deterioration increasing as the drying temperature increases (Doymaz and Pala, 2002; Vega-Galvez et al., 2008).

Some studies cited in the literature have addressed the influence of drying conditions on the quality characteristics of the products (Simal et al., 2005). Conventional drying of pepper causes a major loss of color and texture quality of the final product.

#### 4.3. Antioxidant Activity

An antioxidant is a molecule that inhibits the oxidation of other molecules. Oxidation is a chemical reaction that transfers electrons or hydrogen from a substance to an oxidizing agent. Oxidation reactions can produce free radicals. In turn, these radicals can start chain reactions. When the chain reaction occurs in a cell, it can cause damage or death to the cell. Antioxidants terminate these chain reactions by removing free radical intermediates, and inhibit other oxidation reactions. They do this by being oxidized themselves, so antioxidants are often reducing agents such as thiols, ascorbic acid, or polyphenols.



Figure 4.8. Plot of DPPH radical scavenging activity versus production days

Change of DPPH radical scavenging activity of isot samples are shown in Figure 4.8. In all of the samples (except the industrial sample) there were some fluctuations by time. When comparing the final values there were significant differences in each other. Samples of simulated traditional and industrial methods showed similar fluctuations at the same days. In contrast, considering the industrially simulated samples by process temperatures, antioxidant activity decreased with increasing process temperature.

When comparing the industrial sample with the others, the industrial sample was shown very low antioxidant activity with respect to those of the other isot samples. Reason of this situation could be the decrease in the metabolic activity of industrial sample. It was assumed that industrial sample might have completed its metabolic activity during the production process and so DPPH radical scavenging activity remained constant. In contrast, metabolic reactions in the other samples were continued during the production process because of the lack of industrial production process conditions. Antonio Vega-Galvez et al. (2009) studied the effects of process temperatures between 50 and 90 °C on physicochemical properties, rehydration, color, texture, vitamin C, antioxidant capacity and total phenolics during the drying of red pepper. The radical scavenging activity showed higher antioxidant activity at high temperatures (i.e. 80 and 90 °C) rather than at low temperatures (i.e. 50, 60 and 70 °C). However, in this study it is hard to see a regular decrease or increase in radical scavenging activity, because of lack of necessary conditions for the reactions. The metabolic activities of the sample could have stopped in industrial sample due to the high temperature in the process.

## 4.4. Total Phenolic Content

Phenols, sometimes called phenolics, are a class of chemical compounds consisting of a hydroxyl group(—OH) bonded directly to an aromatic hydrocarbon group. Tota lphenoliccontent (TPC) was estimated as gallic acid equivalents (GAE) as described by Folin–Ciocalteau's (FC) method. Changes in mg gallic acid/gr extract values against to production days were shown in Figure4.9.There is not a significant difference between the initial and final values of TPC (P>0.05 in appendix). There were fluctuations in TPC values of all samples during the production because metabolic activity was expected to continue. Only there is a significant increase in the final value of the simulated traditional sample. It could be due to the insufficient process conditions compared with the industrial process. Maximum increase and the minimum decrease were determined in simulated traditional sample and the sample processed at 25°C, respectively.

Antonio Vega-Galvez et al.(2009) studied total phenolic content during drying of red pepper, a decrease in total phenolic content was determined as air-drying temperature decreased.

#### 4.5. Ascorbic Acid

Vitamin C or L-ascorbic acid, or simply ascorbate (the anion of ascorbic acid), is an essential nutrient for humans and certain other animal species.

Ascorbic acid content increases with pepper fruit ripening while, losses during post harvest handlings (Martinez et al., 2005)



Figure 4.9. Plot of total phenolic content versus production days



Figure 4.10.Plot of ascorbic acid content versus production days

Changes in amount of ascorbic acid of isot samples in production process are shown in Figure 4.10.Therewere a significant decreases in Vitamin C during the production(p<0,05) because isot production is a kind of drying process and vitamins are heat sensitive substances. Inyang and Ike, (1998) reported that dried peppers after all rehydration contained less ascorbic acid than the fresh pepper due to a combination of leaching, oxidation and thermal destruction of ascorbic acid during hot-air drying and subsequent rehydration. The air drying temperature had a detrimental effect on the retention of ascorbic acid, as heated air inherently exposes the products oxidation, reducing their ascorbic acid content. In this study the ascorbic acid amounts were decreased during production similarly because isot production process is a kind of drying process. The maximum and minimum changes in were observed in samples of industrial method and in samples of simulated industrial production at 35°C, respectively.

## 4.6. Non-Enzymatic Browning

Non-enzymatic, or oxidative, browning is a chemical process that produces a brown color in foods without the activity of enzymes. The two main forms of non-enzymatic browning are caramelization and the Maillard reactions. Both vary in reaction rate as a function of water activity.





Changes in non-enzymatic browning (NEB) of isot samples in production process are shown in Figure 4.11. There were a significant increase in absorbance (at 420nm) of isot samples during production (p<0,05 in appendix). Maximum change was in samples produced in industrial and the minimum change is in sample produced with simulated traditional method. The NEB values of all samples have not reached to the value of NEB for samples of the industrial method (factory produced). All methods excluding the industrial method possibly did not provide the necessary processing parameters such as pressure and temperature for the formation of brownish-dark color.

Tannenbaum and Young (1985) said that the decomposition of ascorbic acid was closely tied to the non-enzymic browning in some food products. So, the change of ascorbic acid contents shown in Figure 4.10may be related to the formation of browning products of non-enzymic browning reactions.



## 4.7. pH

Figure 4.12. Plot of pH change versus production days

Changes in pH of isot samples in production process are shown in Figure 4.12. There were significant decreases in pH of isot samples by time and during production (p<0,05 in appendix) because of the acid formation. pH of the fresh pepper was 5.17 and after the production there was a significant decrease in all of the samples. Maximum change from 5.17 to 4.42 was in the sample produced at  $35^{\circ}$ C,minimum change from 5.17 to 4.62 was in the sample of produced at  $45^{\circ}$ C. The pH and titritable acidity values were significantly affected by the drying process (Özgür et al., 2011). However, the difference in pH between all samples were not significant

## 4.8. Acidity

In this study titratable acidity of our isot samples was determined by titration against 0.1 N NaOH and expressed in malic acid equivalents, as the predominant acid in bell

pepper. Vega-Gálvez et al. (2009) also expressed the level of titratable acidity as malic acid in dried Hungarian red peppers.



Figure 4.13. Plot of acidity change versus production days

Some authors expressed titratable acidy in other acids such as citric acid (Özgür et al., 2011).

Changes in acidity of isot samples in production processes are shown in Figure 4.13. There was a similar trend in acidity values by time except industrial sample. The higher TA values were determined in the industrial sample. It is a fact that isot production was tried to be simulated at laboratory conditions, some processing parameters (such as temperature and pressure) were not enough for the isot formation and less metabolic activity rates at low temperatures compared the real isot production.

Luning et al. (1995) investigated the influence of hot-air drying on the composition of both volatile and non-volatile flavour compounds of different Dutch bell pepper cultivars. Glucose, ascorbic, fructose, oxalic and citric acid decreased significantly after drying process, while the levels of sucrose, malic, cis-aconiticand fumaricacid increased. Luning et al. (1995) stated thatglucose, fructose and ascorbic acid probably participate in Maillard reactions during heating (drying). These results support our selection for considering malic acid as the predominant acid.

## 4.9. Capsaicinoid Content

Capsaicin is the main capsaicinoid in peppers, followed by dihydrocapsaicin.and others such as nordihydrocapsaicin, homodihydrocapsaicin and homocapsaicin

The pungency level of capcicum species depends on the concentration of capsainoids; especially capcaicin in the pepper fruit.



Figure 4.14.Plot of capcaicin change versus production days



Figure 4.15. Plot of Dihydrocapcaicin change versus production days

The change of capcaicin and dehydrocapcaicin in industrial sample by time were shown in Figure 4.14. and Figure 4.15., respectively and calculated according to

datas which are shown in appendix. Firstly there was a significant increase in dihydrocapcaicin amount of industrially produced isot sample during production (p<0,05) after that, dihydrocapcaicin values reached a plateau, and stayed almost constant. It could be said that, most metabolic activities have been completed there.

Kurian and Starks (2002) studied quantitative extraction procedure for analysis of capsaicin and dihydrocapsaicin by high-performance liquid chromatographic (HPLC) in whole chili peppers. Relative to dry weight, the orange habañero peppers contained 8840 ppm capsaicin and 3940 ppm dihydrocapsaicin. Our study contained 4459 ppm/ g sample capsaicin and 43.3 ppm/ g sample dihydrocapsaicin.

These dry weight capsaicinoid contents for our samples are much lower than the previously reported (Kurian and Starks (2002)) samples.

## **CHAPTER V**

## CONCLUSION

In this study the analyses and relationships of color, acidity, total phenolic content, ascorbic acid, antioxidant content, pH, non-enzymatic browning, and capsaicin content of isot pepper(simulated industrial methods at 25°C,35°C,45°C,open atmosphere conditions, simulated traditional method and industrial sample(taken from factory)) were examined.

- The moisture content of the material was very high during the initial phase of the drying which resulted in high drying rates due to the higher moisture diffusion. Afterwards, moisture content for all samples were almost constant
- In all the conditions there was a decrease in "L\*", "a\*" and "b\*" between the first day and the last day samples significantly (p<0,05).
- ASTA color values significantly (P < 0.05) changed by drying methods. An increase in ASTA values determined.
- There is an increase in ASTA color values in increasing temperature.
- The industrial sample was shown to have very low antioxidant activity with respect to the other isot samples. Reason of this situation could be the decrease in the metabolic activity of industrial sample
- Antioxidant activity decreased with increasing process temperature when considering the industrially simulated samples by process temperatures.
- There were fluctuations in TPC values of all samples during the production because metabolic activity was going on.
- There is not a significant difference between the initial and final values of TPC ( P>0.05).
- The industrial isot production process conditions provided better isot formation in quality.
- There were a significant decreases in ascorbic acid during the all production methods (p<0,05) and by time.

- There were a significant increase in NEB -absorbance (at 420nm) of isot samples during production (p<0,05). Maximum change was in samples produced in factory (industrial) and the minimum change is in sample produced with simulated traditional method.
- There were significant decreases in pH of isot samples during production
- The higher TA% values were determined in the industrial sample by time.
- Changes in titrable acidity of isot samples were more or less the same in all production processes except the industrial sample. Little change in TA was observed.
- For capsaicin and dihydrocapsaicin changes for industrial sample, firstly there was a significant increase and then, the values have reached plateau, and stayed almost constant.

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## APPENDIX

## HPLC RESULTS

# Day 0 unknown sample



Detector A Ch1 280nm - 320nm

- -----

| Peak# | Ret. Time | Area      | Height  | Area % | Height % |
|-------|-----------|-----------|---------|--------|----------|
| 1     | 0.179     | 1837      | 406     | 0.001  | 0.002    |
| 2     | 0.313     | 1700      | 300     | 0.000  | 0.002    |
| 3     | 0.447     | 2176      | 431     | 0.001  | 0.002    |
| 4     | 0.516     | 2019      | 359     | 0.001  | 0.002    |
| 5     | 0.631     | 2507      | 350     | 0.001  | 0.002    |
| 6     | 0.782     | 1591      | 264     | 0.000  | 0.001    |
| 7     | 1.123     | 2604      | 358     | 0.001  | 0.002    |
| 8     | 1.250     | 1032      | 275     | 0.000  | 0.001    |
| 9     | 1.421     | 1557      | 243     | 0.000  | 0.001    |
| 10    | 1.768     | 1282062   | 180780  | 0.371  | 0.962    |
| 11    | 2.265     | 15097120  | 1095692 | 4.374  | 5.832    |
| 12    | 2.470     | 3579404   | 565815  | 1.037  | 3.012    |
| 13    | 2.819     | 16272256  | 1011855 | 4.715  | 5.386    |
| 14    | 3.212     | 2691601   | 243529  | 0.780  | 1.296    |
| 15    | 3.534     | 2152061   | 143610  | 0.624  | 0.764    |
| 16    | 3.820     | 1574160   | 113596  | 0.456  | 0.605    |
| 17    | 4.400     | 2206757   | 88221   | 0.639  | 0.470    |
| 18    | 4.817     | 645972    | 56891   | 0.187  | 0.303    |
| 19    | 5.101     | 2514969   | 248334  | 0.729  | 1.322    |
| 20    | 5.344     | 3263916   | 228567  | 0.946  | 1.217    |
| 21    | 6.185     | 642336    | 31471   | 0.186  | 0.168    |
| 22    | 6.759     | 1528473   | 88155   | 0.443  | 0.469    |
| 23    | 7.270     | 257476    | 18759   | 0.075  | 0.100    |
| 24    | 7.937     | 1040722   | 39579   | 0.302  | 0.211    |
| 25    | 8.930     | 9571194   | 513167  | 2.773  | 2.731    |
| 26    | 9.561     | 160466168 | 9072229 | 46.495 | 48.288   |
| 27    | 11.815    | 20114     | 1712    | 0.006  | 0.009    |
| 28    | 12.245    | 2142      | 241     | 0.001  | 0.001    |
| 29    | 13.291    | 113024961 | 4765360 | 32.749 | 25.364   |
| 30    | 13.988    | 194243    | 9001    | 0.056  | 0.048    |
| 31    | 14.378    | 1702866   | 86927   | 0.493  | 0.463    |
| 32    | 16.087    | 18480     | 2140    | 0.005  | 0.011    |
| 33    | 16.137    | 32258     | 2311    | 0.009  | 0.012    |

| Peak# | Ret. Time | Area      | Height   | Area %  | Height % |
|-------|-----------|-----------|----------|---------|----------|
| 34    | 17.339    | 1250      | 320      | 0.000   | 0.002    |
| 35    | 17.512    | 9787      | 751      | 0.003   | 0.004    |
| 36    | 18.141    | 223534    | 8466     | 0.065   | 0.045    |
| 37    | 18.754    | 79878     | 2923     | 0.023   | 0.016    |
| 38    | 19.759    | 915308    | 30919    | 0.265   | 0.165    |
| 39    | 20.815    | 3865825   | 113819   | 1.120   | 0.606    |
| 40    | 22.146    | 19241     | 1247     | 0.006   | 0.007    |
| 41    | 22.808    | 43697     | 3158     | 0.013   | 0.017    |
| 42    | 22.931    | 71633     | 3779     | 0.021   | 0.020    |
| 43    | 23.325    | 3817      | 1309     | 0.001   | 0.007    |
| 44    | 23.408    | 6553      | 1385     | 0.002   | 0.007    |
| 45    | 23.501    | 10957     | 1719     | 0.003   | 0.009    |
| 46    | 23.564    | 5576      | 1610     | 0.002   | 0.009    |
| 47    | 23.646    | 23387     | 1768     | 0.007   | 0.009    |
| 48    | 24.042    | 1878      | 403      | 0.001   | 0.002    |
| 49    | 24.692    | 37180     | 2036     | 0.011   | 0.011    |
| 50    | 24.807    | 6564      | 1129     | 0.002   | 0.006    |
| 51    | 24.941    | 1264      | 242      | 0.000   | 0.001    |
| Total |           | 345126064 | 18787909 | 100.000 | 100.000  |

Day 1 unknown sample



## PeakTable

| Peak# | Ret. Time | Area      | Height   | Area % | Height % |
|-------|-----------|-----------|----------|--------|----------|
| 1     | 1.284     | 2758      | 397      | 0.001  | 0.002    |
| 2     | 1.498     | 2569      | 401      | 0.001  | 0.002    |
| 3     | 1.765     | 1588052   | 227669   | 0.334  | 1.012    |
| 4     | 2.273     | 18658797  | 1330846  | 3.927  | 5.913    |
| 5     | 2.467     | 5014082   | 767678   | 1.055  | 3.411    |
| 6     | 2.822     | 25284226  | 1263699  | 5.321  | 5.615    |
| 7     | 3.536     | 3249473   | 203557   | 0.684  | 0.904    |
| 8     | 3.797     | 1806463   | 139427   | 0.380  | 0.619    |
| 9     | 4.411     | 2271008   | 83450    | 0.478  | 0.371    |
| 10    | 4.819     | 996770    | 89372    | 0.210  | 0.397    |
| 11    | 5.101     | 2618706   | 260308   | 0.551  | 1.157    |
| 12    | 5.342     | 3880587   | 276363   | 0.817  | 1.228    |
| 13    | 6.184     | 1157301   | 55056    | 0.244  | 0.245    |
| 14    | 6.761     | 2000677   | 99636    | 0.421  | 0.443    |
| 15    | 7.279     | 446596    | 32758    | 0.094  | 0.146    |
| 16    | 7.934     | 1373208   | 56049    | 0.289  | 0.249    |
| 17    | 8.929     | 14271083  | 758795   | 3.003  | 3.371    |
| 18    | 9.642     | 221345950 | 10239938 | 46.584 | 45.496   |
| 19    | 11.790    | 2552      | 555      | 0.001  | 0.002    |
| 20    | 11.842    | 3488      | 655      | 0.001  | 0.003    |
| 21    | 12.324    | 5138      | 420      | 0.001  | 0.002    |
| 22    | 13.288    | 161755193 | 6369534  | 34.042 | 28.300   |
| 23    | 14.018    | 224945    | 16402    | 0.047  | 0.073    |
| 24    | 17.378    | 1607      | 444      | 0.000  | 0.002    |
| 25    | 17.549    | 7182      | 826      | 0.002  | 0.004    |
| 26    | 17.597    | 5546      | 822      | 0.001  | 0.004    |
| 27    | 18.149    | 351699    | 13187    | 0.074  | 0.059    |
| 28    | 18.729    | 95908     | 4046     | 0.020  | 0.018    |
| 29    | 19.749    | 1074699   | 36674    | 0.226  | 0.163    |
| 30    | 20.808    | 5381137   | 160257   | 1.132  | 0.712    |
| 31    | 21.958    | 7478      | 1809     | 0.002  | 0.008    |
| 32    | 22.228    | 22534     | 1182     | 0.005  | 0.005    |
| 33    | 22.411    | 6511      | 835      | 0.001  | 0.004    |

| Peak# | Ret. Time | Area      | Height   | Area %  | Height % |
|-------|-----------|-----------|----------|---------|----------|
| 34    | 22.904    | 148163    | 4658     | 0.031   | 0.021    |
| 35    | 23.434    | 8728      | 1729     | 0.002   | 0.008    |
| 36    | 23.510    | 6350      | 1670     | 0.001   | 0.007    |
| 37    | 23.592    | 32601     | 1717     | 0.007   | 0.008    |
| 38    | 24.670    | 32652     | 2021     | 0.007   | 0.009    |
| 39    | 24.757    | 9447      | 1507     | 0.002   | 0.007    |
| 40    | 24.820    | 5575      | 1103     | 0.001   | 0.005    |
| Total |           | 475157441 | 22507451 | 100.000 | 100.000  |



Day 2 unknown sample

## PeakTable

Detector A Ch1 280nm - 320nm

| Peak# | Ret. Time | Area      | Height   | Area % | Height % |
|-------|-----------|-----------|----------|--------|----------|
| 1     | 0.194     | 4486      | 450      | 0.001  | 0.002    |
| 2     | 0.412     | 1581      | 226      | 0.000  | 0.001    |
| 3     | 0.595     | 2019      | 169      | 0.000  | 0.001    |
| 4     | 1.060     | 1033      | 284      | 0.000  | 0.001    |
| 5     | 1.315     | 1144      | 225      | 0.000  | 0.001    |
| 6     | 1.766     | 2392122   | 353482   | 0.411  | 1.315    |
| 7     | 2.275     | 31102888  | 1737621  | 5.345  | 6.463    |
| 8     | 2.824     | 23557740  | 1728918  | 4.048  | 6.430    |
| 9     | 3.072     | 6042846   | 642851   | 1.038  | 2.391    |
| 10    | 3.226     | 5985315   | 550763   | 1.028  | 2.048    |
| 11    | 3.525     | 3541633   | 273386   | 0.609  | 1.017    |
| 12    | 3.774     | 3261644   | 200333   | 0.560  | 0.745    |
| 13    | 4.194     | 765234    | 74262    | 0.131  | 0.276    |
| 14    | 4.388     | 1692248   | 85490    | 0.291  | 0.318    |
| 15    | 4.818     | 1402715   | 129205   | 0.241  | 0.481    |
| 16    | 5.100     | 2969447   | 299562   | 0.510  | 1.114    |
| 17    | 5.339     | 4484582   | 348339   | 0.771  | 1.296    |
| 18    | 6.183     | 898596    | 68625    | 0.154  | 0.255    |
| 19    | 6.762     | 1793110   | 100760   | 0.308  | 0.375    |
| 20    | 7.257     | 367489    | 37192    | 0.063  | 0.138    |
| 21    | 7.928     | 860975    | 58977    | 0.148  | 0.219    |
| 22    | 8.480     | 456973    | 23582    | 0.079  | 0.088    |
| 23    | 8.927     | 13511756  | 923397   | 2.322  | 3.434    |
| 24    | 9.442     | 258598274 | 10140467 | 44.436 | 37.715   |
| 25    | 12.230    | 5318      | 565      | 0.001  | 0.002    |
| 26    | 12.593    | 5561      | 868      | 0.001  | 0.003    |
| 27    | 12.665    | 3510      | 811      | 0.001  | 0.003    |
| 28    | 13.285    | 204890093 | 8702826  | 35.207 | 32.368   |
| 29    | 14.035    | 3483296   | 77228    | 0.599  | 0.287    |
| 30    | 17.551    | 15774     | 1171     | 0.003  | 0.004    |
| 31    | 17.654    | 2784      | 863      | 0.000  | 0.003    |
| 32    | 18.146    | 580075    | 21742    | 0.100  | 0.081    |
| 33    | 18.683    | 45508     | 5553     | 0.008  | 0.021    |

| Peak# | Ret. Time | Area      | Height   | Area %  | Height % |
|-------|-----------|-----------|----------|---------|----------|
| 34    | 18.762    | 78789     | 5581     | 0.014   | 0.021    |
| 35    | 19.746    | 1564681   | 53244    | 0.269   | 0.198    |
| 36    | 20.803    | 7322493   | 220276   | 1.258   | 0.819    |
| 37    | 22.082    | 2551      | 416      | 0.000   | 0.002    |
| 38    | 22.881    | 142314    | 4955     | 0.024   | 0.018    |
| 39    | 23.458    | 5399      | 1392     | 0.001   | 0.005    |
| 40    | 23.480    | 2867      | 1455     | 0.000   | 0.005    |
| 41    | 23.517    | 5859      | 1475     | 0.001   | 0.005    |
| 42    | 23.583    | 6261      | 1600     | 0.001   | 0.006    |
| 43    | 23.650    | 15254     | 1621     | 0.003   | 0.006    |
| 44    | 24.683    | 77181     | 3304     | 0.013   | 0.012    |
| 45    | 24.882    | 8668      | 1518     | 0.001   | 0.006    |
| Total |           | 581960087 | 26887029 | 100.000 | 100.000  |

Day 3 unknown sample



## PeakTable

## Detector A Ch1 280nm - 320nm

| Peak# | Ret. Time | Area      | Height   | Area % | Height % |
|-------|-----------|-----------|----------|--------|----------|
| 1     | 0.220     | 1284      | 372      | 0.000  | 0.001    |
| 2     | 0.375     | 1188      | 337      | 0.000  | 0.001    |
| 3     | 0.626     | 12589     | 943      | 0.002  | 0.004    |
| 4     | 0.767     | 3817      | 915      | 0.001  | 0.003    |
| 5     | 0.844     | 9276      | 982      | 0.002  | 0.004    |
| 6     | 1.050     | 12661     | 1233     | 0.002  | 0.005    |
| 7     | 1.190     | 6396      | 1095     | 0.001  | 0.004    |
| 8     | 1.373     | 12653     | 1389     | 0.002  | 0.005    |
| 9     | 1.764     | 2319196   | 333534   | 0.399  | 1.238    |
| 10    | 2.280     | 22764622  | 1576654  | 3.916  | 5.854    |
| 11    | 2.464     | 6156798   | 988291   | 1.059  | 3.670    |
| 12    | 2.823     | 23786072  | 1746417  | 4.092  | 6.485    |
| 13    | 3.072     | 11334633  | 658721   | 1.950  | 2.446    |
| 14    | 3.521     | 3339971   | 251490   | 0.575  | 0.934    |
| 15    | 3.795     | 3598183   | 210242   | 0.619  | 0.781    |
| 16    | 4.197     | 912101    | 86990    | 0.157  | 0.323    |
| 17    | 4.364     | 1948139   | 95848    | 0.335  | 0.356    |
| 18    | 4.816     | 1924279   | 164670   | 0.331  | 0.611    |
| 19    | 5.097     | 3766717   | 365304   | 0.648  | 1.356    |
| 20    | 5.335     | 5586548   | 377431   | 0.961  | 1.401    |
| 21    | 6.177     | 1960705   | 97658    | 0.337  | 0.363    |
| 22    | 6.759     | 2840331   | 127612   | 0.489  | 0.474    |
| 23    | 7.272     | 869596    | 56483    | 0.150  | 0.210    |
| 24    | 7.926     | 2180308   | 87581    | 0.375  | 0.325    |
| 25    | 8.472     | 2002304   | 92474    | 0.344  | 0.343    |
| 26    | 8.925     | 16740872  | 966764   | 2.880  | 3.590    |
| 27    | 9.683     | 261529198 | 10240642 | 44.987 | 38.024   |
| 28    | 11.417    | 14988     | 955      | 0.003  | 0.004    |
| 29    | 12.183    | 6193      | 269      | 0.001  | 0.001    |
| 30    | 12.638    | 2327      | 366      | 0.000  | 0.001    |
| 31    | 13.282    | 191661247 | 8026538  | 32.968 | 29.803   |
| 32    | 14.033    | 3498643   | 67501    | 0.602  | 0.251    |
| 33    | 18.137    | 1580761   | 29808    | 0.272  | 0.111    |

| Peak# | Ret. Time | Area      | Height   | Area %  | Height % |
|-------|-----------|-----------|----------|---------|----------|
| 34    | 19.742    | 1747491   | 53521    | 0.301   | 0.199    |
| 35    | 20.799    | 6949397   | 204553   | 1.195   | 0.760    |
| 36    | 22.431    | 1209      | 393      | 0.000   | 0.001    |
| 37    | 22.835    | 67830     | 5303     | 0.012   | 0.020    |
| 38    | 22.878    | 103170    | 5256     | 0.018   | 0.020    |
| 39    | 23.506    | 28655     | 1625     | 0.005   | 0.006    |
| 40    | 23.827    | 2126      | 611      | 0.000   | 0.002    |
| 41    | 23.893    | 4025      | 569      | 0.001   | 0.002    |
| 42    | 24.630    | 59314     | 2553     | 0.010   | 0.009    |
| Total |           | 581347812 | 26931892 | 100.000 | 100.000  |

Day 4 unknown sample


| Peak# | Ret. Time | Area      | Height   | Area % | Height % |
|-------|-----------|-----------|----------|--------|----------|
| 1     | 0.255     | 1352      | 230      | 0.000  | 0.001    |
| 2     | 0.445     | 1165      | 316      | 0.000  | 0.001    |
| 3     | 0.508     | 1150      | 405      | 0.000  | 0.002    |
| 4     | 0.788     | 9166      | 1020     | 0.002  | 0.004    |
| 5     | 1.334     | 68070     | 3376     | 0.012  | 0.014    |
| 6     | 1.440     | 34800     | 3675     | 0.006  | 0.015    |
| 7     | 1.759     | 2141307   | 308130   | 0.393  | 1.242    |
| 8     | 2.279     | 28133826  | 1545903  | 5.160  | 6.229    |
| 9     | 2.823     | 21460556  | 1577166  | 3.936  | 6.355    |
| 10    | 3.071     | 10443136  | 583255   | 1.915  | 2.350    |
| 11    | 3.531     | 3386160   | 242420   | 0.621  | 0.977    |
| 12    | 3.716     | 834502    | 186187   | 0.153  | 0.750    |
| 13    | 3.783     | 2220686   | 186809   | 0.407  | 0.753    |
| 14    | 4.198     | 796852    | 79747    | 0.146  | 0.321    |
| 15    | 4.373     | 1921142   | 90812    | 0.352  | 0.366    |
| 16    | 4.814     | 1541875   | 134110   | 0.283  | 0.540    |
| 17    | 5.096     | 2986626   | 286839   | 0.548  | 1.156    |
| 18    | 5.335     | 4328375   | 294582   | 0.794  | 1.187    |
| 19    | 6.176     | 1537451   | 74975    | 0.282  | 0.302    |
| 20    | 6.757     | 2515148   | 111074   | 0.461  | 0.448    |
| 21    | 7.303     | 554990    | 40316    | 0.102  | 0.162    |
| 22    | 7.934     | 2034028   | 81037    | 0.373  | 0.327    |
| 23    | 8.561     | 1597795   | 90221    | 0.293  | 0.364    |
| 24    | 8.926     | 16089536  | 912022   | 2.951  | 3.675    |
| 25    | 9.675     | 253534721 | 10237962 | 46.497 | 41.252   |
| 26    | 12.612    | 2554      | 439      | 0.000  | 0.002    |
| 27    | 13.284    | 175557982 | 7406634  | 32.197 | 29.843   |
| 28    | 14.035    | 2911402   | 60386    | 0.534  | 0.243    |
| 29    | 17.073    | 1228      | 212      | 0.000  | 0.001    |
| 30    | 17.520    | 16672     | 1153     | 0.003  | 0.005    |
| 31    | 17.681    | 4306      | 985      | 0.001  | 0.004    |
| 32    | 18.138    | 493688    | 18682    | 0.091  | 0.075    |
| 33    | 18.702    | 152129    | 5681     | 0.028  | 0.023    |

| Peak# | Ret. Time | Area      | Height   | Area %  | Height % |
|-------|-----------|-----------|----------|---------|----------|
| 34    | 19.746    | 1384037   | 45913    | 0.254   | 0.185    |
| 35    | 20.797    | 6329816   | 189436   | 1.161   | 0.763    |
| 36    | 22.186    | 1608      | 314      | 0.000   | 0.001    |
| 37    | 22.904    | 124273    | 4457     | 0.023   | 0.018    |
| 38    | 23.327    | 8378      | 1183     | 0.002   | 0.005    |
| 39    | 23.521    | 13721     | 1668     | 0.003   | 0.007    |
| 40    | 23.613    | 7987      | 1436     | 0.001   | 0.006    |
| 41    | 23.716    | 9824      | 1197     | 0.002   | 0.005    |
| 42    | 24.625    | 50395     | 3281     | 0.009   | 0.013    |
| 43    | 24.700    | 24280     | 2695     | 0.004   | 0.011    |
| Total |           | 545268694 | 24818340 | 100.000 | 100.000  |

Day 5 unknown sample



| Peak# | Ret. Time | Area      | Height  | Area % | Height % |
|-------|-----------|-----------|---------|--------|----------|
| 1     | 0.292     | 6959      | 795     | 0.002  | 0.004    |
| 2     | 0.404     | 7851      | 1018    | 0.002  | 0.005    |
| 3     | 0.658     | 19405     | 1811    | 0.005  | 0.009    |
| 4     | 1.184     | 113627    | 4654    | 0.031  | 0.022    |
| 5     | 1.413     | 59823     | 5121    | 0.016  | 0.025    |
| 6     | 1.824     | 2371111   | 296079  | 0.644  | 1.420    |
| 7     | 2.319     | 14449894  | 1151399 | 3.927  | 5.522    |
| 8     | 2.485     | 18843151  | 1278596 | 5.121  | 6.132    |
| 9     | 2.820     | 28640007  | 2276179 | 7.783  | 10.916   |
| 10    | 3.095     | 11299375  | 905789  | 3.071  | 4.344    |
| 11    | 3.644     | 9806229   | 640044  | 2.665  | 3.070    |
| 12    | 3.965     | 5485989   | 385463  | 1.491  | 1.849    |
| 13    | 4.486     | 2079728   | 117872  | 0.565  | 0.565    |
| 14    | 4.815     | 3097861   | 235276  | 0.842  | 1.128    |
| 15    | 5.068     | 659909    | 69892   | 0.179  | 0.335    |
| 16    | 5.333     | 1476388   | 56861   | 0.401  | 0.273    |
| 17    | 6.071     | 570555    | 28186   | 0.155  | 0.135    |
| 18    | 6.548     | 1056057   | 55443   | 0.287  | 0.266    |
| 19    | 6.900     | 2722950   | 81232   | 0.740  | 0.390    |
| 20    | 7.947     | 1468394   | 49316   | 0.399  | 0.237    |
| 21    | 8.472     | 1243896   | 50007   | 0.338  | 0.240    |
| 22    | 8.927     | 9127776   | 509288  | 2.481  | 2.442    |
| 23    | 9.556     | 153536353 | 8604202 | 41.726 | 41.264   |
| 24    | 11.778    | 54483     | 3732    | 0.015  | 0.018    |
| 25    | 12.645    | 1003      | 230     | 0.000  | 0.001    |
| 26    | 13.283    | 90536866  | 3693896 | 24.605 | 17.715   |
| 27    | 14.020    | 323352    | 20653   | 0.088  | 0.099    |
| 28    | 14.320    | 1193840   | 57930   | 0.324  | 0.278    |
| 29    | 15.471    | 14877     | 604     | 0.004  | 0.003    |
| 30    | 16.121    | 1092      | 190     | 0.000  | 0.001    |
| 31    | 16.432    | 59499     | 2102    | 0.016  | 0.010    |
| 32    | 16.949    | 17730     | 1607    | 0.005  | 0.008    |
| 33    | 17.081    | 8875      | 1505    | 0.002  | 0.007    |

| Peak# | Ret. Time | Area      | Height   | Area %  | Height % |
|-------|-----------|-----------|----------|---------|----------|
| 34    | 17.494    | 56783     | 3764     | 0.015   | 0.018    |
| 35    | 17.542    | 38657     | 3920     | 0.011   | 0.019    |
| 36    | 18.156    | 3296332   | 124787   | 0.896   | 0.598    |
| 37    | 19.739    | 428525    | 16868    | 0.116   | 0.081    |
| 38    | 20.796    | 3690328   | 103224   | 1.003   | 0.495    |
| 39    | 22.782    | 12478     | 1552     | 0.003   | 0.007    |
| 40    | 22.918    | 34386     | 1945     | 0.009   | 0.009    |
| 41    | 23.346    | 1308      | 252      | 0.000   | 0.001    |
| 42    | 23.489    | 3030      | 543      | 0.001   | 0.003    |
| 43    | 23.546    | 1243      | 518      | 0.000   | 0.002    |
| 44    | 23.669    | 6376      | 1038     | 0.002   | 0.005    |
| 45    | 23.742    | 8637      | 1071     | 0.002   | 0.005    |
| 46    | 23.939    | 5865      | 757      | 0.002   | 0.004    |
| 47    | 24.112    | 1798      | 427      | 0.000   | 0.002    |
| 48    | 24.268    | 1721      | 358      | 0.000   | 0.002    |
| 49    | 24.364    | 2081      | 468      | 0.001   | 0.002    |
| 50    | 24.467    | 2564      | 680      | 0.001   | 0.003    |
| 51    | 24.614    | 7365      | 792      | 0.002   | 0.004    |
| 52    | 24.680    | 4147      | 821      | 0.001   | 0.004    |
| 53    | 24.789    | 4784      | 708      | 0.001   | 0.003    |
| Total |           | 367963318 | 20851463 | 100.000 | 100.000  |

Day 0 unknown sample+capsaicin (50ppm)



| Peak# | Ret. Time | Area      | Height   | Area % | Height % |
|-------|-----------|-----------|----------|--------|----------|
| 1     | 0.206     | 4576      | 510      | 0.001  | 0.003    |
| 2     | 0.813     | 1156      | 132      | 0.000  | 0.001    |
| 3     | 1.095     | 1299      | 438      | 0.000  | 0.003    |
| 4     | 1.310     | 1619      | 288      | 0.000  | 0.002    |
| 5     | 1.754     | 833857    | 123846   | 0.221  | 0.739    |
| 6     | 2.272     | 14296109  | 843832   | 3.785  | 5.034    |
| 7     | 2.820     | 12723207  | 660972   | 3.369  | 3.943    |
| 8     | 3.545     | 1363083   | 98612    | 0.361  | 0.588    |
| 9     | 3.837     | 1331790   | 89424    | 0.353  | 0.534    |
| 10    | 4.410     | 1260208   | 50172    | 0.334  | 0.299    |
| 11    | 4.855     | 539239    | 48769    | 0.143  | 0.291    |
| 12    | 5.141     | 1588497   | 157333   | 0.421  | 0.939    |
| 13    | 5.389     | 2072998   | 153609   | 0.549  | 0.916    |
| 14    | 6.237     | 504768    | 23114    | 0.134  | 0.138    |
| 15    | 6.841     | 1418964   | 67181    | 0.376  | 0.401    |
| 16    | 8.084     | 1491116   | 45463    | 0.395  | 0.271    |
| 17    | 9.107     | 10041496  | 599891   | 2.659  | 3.579    |
| 18    | 9.875     | 245517460 | 10245135 | 65.006 | 61.124   |
| 19    | 12.624    | 1411      | 351      | 0.000  | 0.002    |
| 20    | 12.930    | 11301     | 1076     | 0.003  | 0.006    |
| 21    | 13.617    | 73036288  | 3275422  | 19.338 | 19.542   |
| 22    | 14.580    | 5546489   | 131463   | 1.469  | 0.784    |
| 23    | 16.474    | 75620     | 3456     | 0.020  | 0.021    |
| 24    | 16.747    | 22945     | 2094     | 0.006  | 0.012    |
| 25    | 17.015    | 4593      | 878      | 0.001  | 0.005    |
| 26    | 17.111    | 3652      | 900      | 0.001  | 0.005    |
| 27    | 17.200    | 4691      | 587      | 0.001  | 0.004    |
| 28    | 17.742    | 3684      | 539      | 0.001  | 0.003    |
| 29    | 17.804    | 1671      | 598      | 0.000  | 0.004    |
| 30    | 17.875    | 3663      | 808      | 0.001  | 0.005    |
| 31    | 18.017    | 8239      | 962      | 0.002  | 0.006    |
| 32    | 18.144    | 4792      | 691      | 0.001  | 0.004    |
| 33    | 18.248    | 2981      | 765      | 0.001  | 0.005    |

| Peak# | Ret. Time | Area      | Height   | Area %  | Height % |
|-------|-----------|-----------|----------|---------|----------|
| 34    | 18.637    | 167089    | 6448     | 0.044   | 0.038    |
| 35    | 19.023    | 6272      | 1813     | 0.002   | 0.011    |
| 36    | 19.104    | 11109     | 1953     | 0.003   | 0.012    |
| 37    | 19.319    | 66317     | 2691     | 0.018   | 0.016    |
| 38    | 20.300    | 674340    | 21407    | 0.179   | 0.128    |
| 39    | 21.412    | 2851941   | 79097    | 0.755   | 0.472    |
| 40    | 22.706    | 1459      | 308      | 0.000   | 0.002    |
| 41    | 22.807    | 1137      | 345      | 0.000   | 0.002    |
| 42    | 22.855    | 1413      | 288      | 0.000   | 0.002    |
| 43    | 22.953    | 2016      | 300      | 0.001   | 0.002    |
| 44    | 23.116    | 2262      | 453      | 0.001   | 0.003    |
| 45    | 23.589    | 45248     | 2793     | 0.012   | 0.017    |
| 46    | 23.625    | 47505     | 2816     | 0.013   | 0.017    |
| 47    | 23.931    | 17829     | 2647     | 0.005   | 0.016    |
| 48    | 24.038    | 18705     | 2607     | 0.005   | 0.016    |
| 49    | 24.175    | 13763     | 2368     | 0.004   | 0.014    |
| 50    | 24.283    | 11114     | 1946     | 0.003   | 0.012    |
| 51    | 24.383    | 20759     | 1727     | 0.005   | 0.010    |
| Total |           | 377683742 | 16761321 | 100.000 | 100.000  |

Day 1 unknown sample+capsaicin (50ppm)



| Peak# | Ret. Time | Area      | Height   | Area % | Height % |
|-------|-----------|-----------|----------|--------|----------|
| 1     | 0.312     | 1647168   | 61705    | 0.397  | 0.338    |
| 2     | 1.750     | 931453    | 141466   | 0.225  | 0.77     |
| 3     | 2.283     | 17395012  | 951177   | 4.197  | 5.21     |
| 4     | 2.823     | 10600799  | 768904   | 2.558  | 4.21     |
| 5     | 3.081     | 4716227   | 264843   | 1.138  | 1.45     |
| 6     | 3.549     | 1727363   | 124722   | 0.417  | 0.684    |
| 7     | 3.825     | 1484624   | 95418    | 0.358  | 0.52     |
| 8     | 4.405     | 1041738   | 37852    | 0.251  | 0.20     |
| 9     | 4.852     | 723761    | 69146    | 0.175  | 0.37     |
| 10    | 5.136     | 1477789   | 149429   | 0.357  | 0.81     |
| 11    | 5.383     | 2141494   | 165107   | 0.517  | 0.90     |
| 12    | 5.928     | 3094      | 605      | 0.001  | 0.00     |
| 13    | 6.231     | 489572    | 31767    | 0.118  | 0.17     |
| 14    | 6.829     | 1251149   | 61984    | 0.302  | 0.34     |
| 15    | 8.087     | 1039143   | 47420    | 0.251  | 0.26     |
| 16    | 8.629     | 3498      | 1404     | 0.001  | 0.00     |
| 17    | 8.717     | 6257      | 862      | 0.002  | 0.00     |
| 18    | 9.105     | 9975528   | 677330   | 2.407  | 3.71     |
| 19    | 9.642     | 256879252 | 10228080 | 61.975 | 56.08    |
| 20    | 11.658    | 1336      | 424      | 0.000  | 0.00     |
| 21    | 11.717    | 8365      | 901      | 0.002  | 0.00     |
| 22    | 12.717    | 3351      | 673      | 0.001  | 0.00     |
| 23    | 12.857    | 25667     | 1622     | 0.006  | 0.00     |
| 24    | 13.613    | 89263723  | 4023842  | 21.536 | 22.06    |
| 25    | 14.448    | 6507368   | 157369   | 1.570  | 0.86     |
| 26    | 16.431    | 26864     | 1935     | 0.006  | 0.01     |
| 27    | 16.553    | 8461      | 2002     | 0.002  | 0.01     |
| 28    | 16.647    | 21679     | 2041     | 0.005  | 0.01     |
| 29    | 16.818    | 17710     | 1551     | 0.004  | 0.00     |
| 30    | 17.163    | 2478      | 541      | 0.001  | 0.00     |
| 31    | 17.235    | 3163      | 534      | 0.001  | 0.00     |
| 32    | 17.673    | 1074      | 182      | 0.000  | 0.00     |
| 33    | 17.763    | 1685      | 256      | 0.000  | 0.00     |

| Peak# | Ret. Time | Area      | Height   | Area %  | Height % |
|-------|-----------|-----------|----------|---------|----------|
| 34    | 17.965    | 1138      | 281      | 0.000   | 0.002    |
| 35    | 18.036    | 1058      | 271      | 0.000   | 0.001    |
| 36    | 18.618    | 267713    | 9171     | 0.065   | 0.050    |
| 37    | 19.050    | 14617     | 4216     | 0.004   | 0.023    |
| 38    | 19.104    | 16557     | 4217     | 0.004   | 0.023    |
| 39    | 19.255    | 110479    | 4568     | 0.027   | 0.025    |
| 40    | 20.308    | 723529    | 23755    | 0.175   | 0.130    |
| 41    | 21.406    | 3676616   | 104199   | 0.887   | 0.571    |
| 42    | 23.158    | 1595      | 713      | 0.000   | 0.004    |
| 43    | 23.231    | 8933      | 1905     | 0.002   | 0.010    |
| 44    | 23.396    | 57142     | 4748     | 0.014   | 0.026    |
| 45    | 23.516    | 179497    | 6059     | 0.043   | 0.033    |
| Total |           | 414486719 | 18237197 | 100.000 | 100.000  |

Day 2 unknown sample+capsaicin (50ppm)



| Peak# | Ret. Time | Area      | Height   | Area % | Height % |
|-------|-----------|-----------|----------|--------|----------|
| 1     | 0.668     | 2849      | 509      | 0.001  | 0.002    |
| 2     | 0.787     | 1207      | 400      | 0.000  | 0.002    |
| 3     | 1.096     | 1836      | 421      | 0.000  | 0.002    |
| 4     | 1.350     | 1845      | 248      | 0.000  | 0.001    |
| 5     | 1.440     | 1923      | 366      | 0.000  | 0.002    |
| 6     | 1.751     | 1411689   | 218717   | 0.290  | 1.027    |
| 7     | 2.285     | 23637258  | 1272003  | 4.857  | 5.975    |
| 8     | 2.824     | 14228506  | 1064697  | 2.924  | 5.002    |
| 9     | 3.083     | 4379130   | 402694   | 0.900  | 1.892    |
| 10    | 3.254     | 3340316   | 318735   | 0.686  | 1.497    |
| 11    | 3.529     | 2296036   | 169797   | 0.472  | 0.798    |
| 12    | 3.820     | 2083224   | 139025   | 0.428  | 0.653    |
| 13    | 4.213     | 447882    | 42357    | 0.092  | 0.199    |
| 14    | 4.378     | 409479    | 42243    | 0.084  | 0.198    |
| 15    | 4.492     | 433570    | 40172    | 0.089  | 0.189    |
| 16    | 4.848     | 1167477   | 110781   | 0.240  | 0.520    |
| 17    | 5.131     | 1754731   | 177472   | 0.361  | 0.834    |
| 18    | 5.376     | 2754890   | 209859   | 0.566  | 0.986    |
| 19    | 6.222     | 863524    | 54800    | 0.177  | 0.257    |
| 20    | 6.818     | 1825781   | 78437    | 0.375  | 0.368    |
| 21    | 8.073     | 1514692   | 68248    | 0.311  | 0.321    |
| 22    | 8.682     | 12690     | 1259     | 0.003  | 0.006    |
| 23    | 9.101     | 12395135  | 842576   | 2.547  | 3.958    |
| 24    | 9.625     | 274691438 | 10150808 | 56.445 | 47.685   |
| 25    | 12.948    | 73315     | 4535     | 0.015  | 0.021    |
| 26    | 13.605    | 120255462 | 5425816  | 24.711 | 25.488   |
| 27    | 14.444    | 9028863   | 232691   | 1.855  | 1.093    |
| 28    | 16.258    | 1397      | 364      | 0.000  | 0.002    |
| 29    | 16.478    | 1317      | 303      | 0.000  | 0.001    |
| 30    | 17.396    | 1183      | 235      | 0.000  | 0.001    |
| 31    | 18.608    | 84461     | 7707     | 0.017  | 0.036    |
| 32    | 18.657    | 100299    | 7679     | 0.021  | 0.036    |
| 33    | 19.460    | 246090    | 9171     | 0.051  | 0.043    |

| Peak# | Ret. Time | Area      | Height   | Area %  | Height % |
|-------|-----------|-----------|----------|---------|----------|
| 34    | 20.279    | 1681658   | 42472    | 0.346   | 0.200    |
| 35    | 21.387    | 5381625   | 142282   | 1.106   | 0.668    |
| 36    | 22.543    | 1282      | 263      | 0.000   | 0.001    |
| 37    | 22.835    | 1613      | 232      | 0.000   | 0.001    |
| 38    | 23.499    | 101748    | 3325     | 0.021   | 0.016    |
| 39    | 23.883    | 22854     | 2194     | 0.005   | 0.010    |
| 40    | 24.106    | 8872      | 1126     | 0.002   | 0.005    |
| 41    | 24.287    | 1127      | 367      | 0.000   | 0.002    |
| Total |           | 486650271 | 21287386 | 100.000 | 100.000  |

Day 3 unknown sample+capsaicin (50ppm)



| Peak# | Ret. Time | Area      | Height   | Area % | Height % |
|-------|-----------|-----------|----------|--------|----------|
| 1     | 0.768     | 2076064   | 61090    | 0.434  | 0.299    |
| 2     | 1.749     | 1294656   | 202542   | 0.271  | 0.991    |
| 3     | 2.286     | 21957742  | 1150321  | 4.589  | 5.630    |
| 4     | 2.823     | 14110359  | 1073097  | 2.949  | 5.252    |
| 5     | 3.082     | 7484262   | 412687   | 1.564  | 2.020    |
| 6     | 3.526     | 2030411   | 152484   | 0.424  | 0.746    |
| 7     | 3.821     | 2085244   | 138794   | 0.436  | 0.679    |
| 8     | 4.216     | 438259    | 41568    | 0.092  | 0.203    |
| 9     | 4.369     | 801219    | 42420    | 0.167  | 0.208    |
| 10    | 4.846     | 1275525   | 120413   | 0.267  | 0.589    |
| 11    | 5.129     | 1998048   | 200343   | 0.418  | 0.981    |
| 12    | 5.372     | 2817025   | 212226   | 0.589  | 1.039    |
| 13    | 5.944     | 3167      | 554      | 0.001  | 0.003    |
| 14    | 6.219     | 810951    | 54427    | 0.169  | 0.266    |
| 15    | 6.818     | 1674407   | 75753    | 0.350  | 0.371    |
| 16    | 8.073     | 1515191   | 65953    | 0.317  | 0.323    |
| 17    | 8.697     | 8353      | 1164     | 0.002  | 0.006    |
| 18    | 9.099     | 11868203  | 802311   | 2.480  | 3.927    |
| 19    | 9.625     | 278197455 | 10196241 | 58.136 | 49.905   |
| 20    | 12.870    | 9685      | 1056     | 0.002  | 0.005    |
| 21    | 13.603    | 111069176 | 4988448  | 23.211 | 24.416   |
| 22    | 14.473    | 8596756   | 220372   | 1.797  | 1.079    |
| 23    | 16.251    | 3109      | 413      | 0.001  | 0.002    |
| 24    | 16.357    | 1454      | 699      | 0.000  | 0.003    |
| 25    | 16.667    | 24474     | 1648     | 0.005  | 0.008    |
| 26    | 16.733    | 12239     | 1626     | 0.003  | 0.008    |
| 27    | 16.902    | 10945     | 1514     | 0.002  | 0.007    |
| 28    | 17.079    | 7124      | 1211     | 0.001  | 0.006    |
| 29    | 17.113    | 5289      | 1222     | 0.001  | 0.006    |
| 30    | 17.261    | 9648      | 1382     | 0.002  | 0.007    |
| 31    | 17.358    | 12949     | 1333     | 0.003  | 0.007    |
| 32    | 17.632    | 14114     | 1440     | 0.003  | 0.007    |
| 33    | 17.683    | 2657      | 1339     | 0.001  | 0.007    |

| Peak# | Ret. Time | Area      | Height   | Area %  | Height % |
|-------|-----------|-----------|----------|---------|----------|
| 34    | 17.740    | 12801     | 1411     | 0.003   | 0.007    |
| 35    | 17.876    | 5471      | 1617     | 0.001   | 0.008    |
| 36    | 17.942    | 10227     | 1560     | 0.002   | 0.008    |
| 37    | 18.059    | 7942      | 1514     | 0.002   | 0.007    |
| 38    | 18.147    | 4693      | 1672     | 0.001   | 0.008    |
| 39    | 18.586    | 368205    | 12388    | 0.077   | 0.061    |
| 40    | 19.055    | 13414     | 4543     | 0.003   | 0.022    |
| 41    | 19.277    | 185134    | 6230     | 0.039   | 0.030    |
| 42    | 19.780    | 12049     | 2551     | 0.003   | 0.012    |
| 43    | 20.282    | 908359    | 30270    | 0.190   | 0.148    |
| 44    | 21.386    | 4326388   | 124976   | 0.904   | 0.612    |
| 45    | 23.109    | 1962      | 454      | 0.000   | 0.002    |
| 46    | 23.261    | 21751     | 2715     | 0.005   | 0.013    |
| 47    | 23.375    | 23175     | 4627     | 0.005   | 0.023    |
| 48    | 23.508    | 397841    | 6731     | 0.083   | 0.033    |
| Total |           | 478525568 | 20431353 | 100.000 | 100.000  |

Day 4 unknown sample+capsaicin (50ppm)



| Peak# | Ret. Time | Area      | Height   | Area % | Height % |
|-------|-----------|-----------|----------|--------|----------|
| 1     | 0.104     | 1542      | 292      | 0.000  | 0.001    |
| 2     | 0.202     | 1895      | 385      | 0.000  | 0.002    |
| 3     | 0.415     | 1298      | 301      | 0.000  | 0.002    |
| 4     | 1.063     | 35549     | 2512     | 0.008  | 0.013    |
| 5     | 1.155     | 42037     | 2512     | 0.009  | 0.013    |
| 6     | 1.476     | 3818      | 761      | 0.001  | 0.004    |
| 7     | 1.747     | 1180818   | 184941   | 0.262  | 0.949    |
| 8     | 2.288     | 20786318  | 1107861  | 4.611  | 5.687    |
| 9     | 2.822     | 12304273  | 950749   | 2.729  | 4.880    |
| 10    | 3.082     | 6625555   | 356641   | 1.470  | 1.831    |
| 11    | 3.540     | 2066401   | 141474   | 0.458  | 0.726    |
| 12    | 3.820     | 1736877   | 119079   | 0.385  | 0.611    |
| 13    | 4.221     | 378392    | 37525    | 0.084  | 0.193    |
| 14    | 4.373     | 766427    | 38907    | 0.170  | 0.200    |
| 15    | 4.844     | 1039457   | 98425    | 0.231  | 0.505    |
| 16    | 5.126     | 1542614   | 156703   | 0.342  | 0.804    |
| 17    | 5.372     | 2098481   | 162910   | 0.465  | 0.836    |
| 18    | 6.215     | 599542    | 40220    | 0.133  | 0.206    |
| 19    | 6.814     | 1432730   | 66141    | 0.318  | 0.340    |
| 20    | 8.072     | 1340836   | 60619    | 0.297  | 0.311    |
| 21    | 8.642     | 9855      | 1876     | 0.002  | 0.010    |
| 22    | 9.100     | 11026538  | 751598   | 2.446  | 3.858    |
| 23    | 9.633     | 268894790 | 10192354 | 59.647 | 52.320   |
| 24    | 12.491    | 3058      | 643      | 0.001  | 0.003    |
| 25    | 12.601    | 5129      | 1055     | 0.001  | 0.005    |
| 26    | 12.939    | 65527     | 4022     | 0.015  | 0.021    |
| 27    | 13.603    | 101248880 | 4571060  | 22.459 | 23.464   |
| 28    | 14.453    | 7899010   | 200060   | 1.752  | 1.027    |
| 29    | 16.182    | 2275      | 546      | 0.001  | 0.003    |
| 30    | 16.431    | 8669      | 985      | 0.002  | 0.005    |
| 31    | 16.524    | 4835      | 1094     | 0.001  | 0.006    |
| 32    | 16.617    | 6352      | 1288     | 0.001  | 0.007    |
| 33    | 16.774    | 16039     | 1781     | 0.004  | 0.009    |

| Peak# | Ret. Time | Area      | Height   | Area %  | Height % |
|-------|-----------|-----------|----------|---------|----------|
| 34    | 17.029    | 29977     | 2855     | 0.007   | 0.015    |
| 35    | 17.153    | 33542     | 3318     | 0.007   | 0.017    |
| 36    | 17.319    | 74867     | 3456     | 0.017   | 0.018    |
| 37    | 17.763    | 6246      | 1038     | 0.001   | 0.005    |
| 38    | 18.589    | 340654    | 11452    | 0.076   | 0.059    |
| 39    | 19.286    | 253336    | 7573     | 0.056   | 0.039    |
| 40    | 19.702    | 59098     | 8030     | 0.013   | 0.041    |
| 41    | 20.273    | 1445447   | 37375    | 0.321   | 0.192    |
| 42    | 21.385    | 4812828   | 127354   | 1.068   | 0.654    |
| 43    | 23.854    | 380429    | 9037     | 0.084   | 0.046    |
| 44    | 23.989    | 161670    | 7973     | 0.036   | 0.041    |
| 45    | 24.393    | 32223     | 3432     | 0.007   | 0.018    |
| 46    | 24.679    | 4512      | 719      | 0.001   | 0.004    |
| Total |           | 450810643 | 19480929 | 100.000 | 100.000  |

Day 5 unknown sample+capsaicin (50ppm)



| Detector A ( | Ch1 280 | nm - 320nm |
|--------------|---------|------------|
|--------------|---------|------------|

| Peak# | Ret. Time | Area      | Height   | Area % | Height % |
|-------|-----------|-----------|----------|--------|----------|
| 1     | 0.122     | 1602      | 362      | 0.000  | 0.002    |
| 2     | 0.738     | 2192      | 437      | 0.001  | 0.002    |
| 3     | 0.814     | 1113      | 257      | 0.000  | 0.001    |
| 4     | 0.980     | 2573      | 600      | 0.001  | 0.003    |
| 5     | 1.170     | 6885      | 701      | 0.002  | 0.004    |
| 6     | 1.345     | 6091      | 691      | 0.002  | 0.004    |
| 7     | 1.469     | 3908      | 785      | 0.001  | 0.004    |
| 8     | 1.784     | 1218373   | 178474   | 0.314  | 0.978    |
| 9     | 2.320     | 10149789  | 781523   | 2.615  | 4.282    |
| 10    | 2.485     | 10991834  | 802878   | 2.832  | 4.399    |
| 11    | 2.820     | 17083128  | 1344567  | 4.402  | 7.367    |
| 12    | 3.100     | 6878613   | 548633   | 1.772  | 3.006    |
| 13    | 3.474     | 841762    | 118904   | 0.217  | 0.651    |
| 14    | 3.662     | 5221103   | 377328   | 1.345  | 2.067    |
| 15    | 3.983     | 3040518   | 227772   | 0.783  | 1.248    |
| 16    | 4.510     | 1170159   | 69024    | 0.301  | 0.378    |
| 17    | 4.843     | 2121264   | 173204   | 0.547  | 0.949    |
| 18    | 5.105     | 380537    | 38263    | 0.098  | 0.210    |
| 19    | 5.363     | 704564    | 31672    | 0.182  | 0.174    |
| 20    | 6.168     | 351866    | 15627    | 0.091  | 0.086    |
| 21    | 6.597     | 732627    | 36656    | 0.189  | 0.201    |
| 22    | 6.795     | 411050    | 38749    | 0.106  | 0.212    |
| 23    | 7.009     | 1266581   | 47047    | 0.326  | 0.258    |
| 24    | 8.076     | 1255856   | 38331    | 0.324  | 0.210    |
| 25    | 9.099     | 10276085  | 591815   | 2.648  | 3.243    |
| 26    | 9.875     | 251832074 | 10244686 | 64.886 | 56.133   |
| 27    | 12.877    | 3022      | 427      | 0.001  | 0.002    |
| 28    | 13.602    | 54485780  | 2208172  | 14.039 | 12.099   |
| 29    | 14.504    | 1088011   | 39123    | 0.280  | 0.214    |
| 30    | 15.558    | 1356      | 317      | 0.000  | 0.002    |
| 31    | 15.776    | 40326     | 2456     | 0.010  | 0.013    |
| 32    | 16.385    | 35486     | 2651     | 0.009  | 0.015    |
| 33    | 16.547    | 25319     | 3310     | 0.007  | 0.018    |

| Peak# | Ret. Time | Area      | Height   | Area %  | Height % |
|-------|-----------|-----------|----------|---------|----------|
| 34    | 16.639    | 19381     | 3676     | 0.005   | 0.020    |
| 35    | 16.770    | 40307     | 4249     | 0.010   | 0.023    |
| 36    | 16.848    | 20278     | 4086     | 0.005   | 0.022    |
| 37    | 16.945    | 22683     | 4262     | 0.006   | 0.023    |
| 38    | 17.116    | 36741     | 4549     | 0.009   | 0.025    |
| 39    | 17.252    | 42380     | 5315     | 0.011   | 0.029    |
| 40    | 17.308    | 21161     | 5300     | 0.005   | 0.029    |
| 41    | 17.382    | 24659     | 5473     | 0.006   | 0.030    |
| 42    | 17.620    | 79497     | 6886     | 0.020   | 0.038    |
| 43    | 17.783    | 74135     | 7974     | 0.019   | 0.044    |
| 44    | 18.436    | 3158127   | 85983    | 0.814   | 0.471    |
| 45    | 20.274    | 737185    | 19968    | 0.190   | 0.109    |
| 46    | 21.377    | 1532040   | 49273    | 0.395   | 0.270    |
| 47    | 22.180    | 39443     | 5060     | 0.010   | 0.028    |
| 48    | 22.671    | 170631    | 7126     | 0.044   | 0.039    |
| 49    | 22.751    | 37295     | 6913     | 0.010   | 0.038    |
| 50    | 22.863    | 36174     | 6604     | 0.009   | 0.036    |
| 51    | 22.917    | 40902     | 6446     | 0.011   | 0.035    |
| 52    | 23.040    | 32616     | 6076     | 0.008   | 0.033    |
| 53    | 23.121    | 30518     | 5746     | 0.008   | 0.031    |
| 54    | 23.242    | 31406     | 5314     | 0.008   | 0.029    |
| 55    | 23.325    | 12877     | 5193     | 0.003   | 0.028    |
| 56    | 23.411    | 30720     | 5217     | 0.008   | 0.029    |
| 57    | 23.487    | 77647     | 5184     | 0.020   | 0.028    |
| 58    | 23.736    | 58773     | 4193     | 0.015   | 0.023    |
| 59    | 24.020    | 35384     | 3143     | 0.009   | 0.017    |
| 60    | 24.225    | 16319     | 2017     | 0.004   | 0.011    |
| 61    | 24.418    | 15033     | 1513     | 0.004   | 0.008    |
| 62    | 24.592    | 2613      | 926      | 0.001   | 0.005    |
| 63    | 24.633    | 2940      | 816      | 0.001   | 0.004    |
| 64    | 24.767    | 3393      | 567      | 0.001   | 0.003    |
| 65    | 24.838    | 2060      | 374      | 0.001   | 0.002    |
| Total |           | 388116765 | 18250864 | 100.000 | 100.000  |

Day 0 unknown sample+dihydrocapsaicin (50ppm)



| Peak# | Ret. Time | Area      | Height   | Area % | Height % |
|-------|-----------|-----------|----------|--------|----------|
| 1     | 0.332     | 2022      | 399      | 0.000  | 0.002    |
| 2     | 0.531     | 2747      | 370      | 0.001  | 0.002    |
| 3     | 0.657     | 1023      | 408      | 0.000  | 0.002    |
| 4     | 0.758     | 1117      | 296      | 0.000  | 0.001    |
| 5     | 0.851     | 1098      | 264      | 0.000  | 0.001    |
| 6     | 1.024     | 1013      | 304      | 0.000  | 0.002    |
| 7     | 1.171     | 2028      | 379      | 0.000  | 0.002    |
| 8     | 1.452     | 4266      | 608      | 0.001  | 0.003    |
| 9     | 1.726     | 750353    | 113750   | 0.158  | 0.563    |
| 10    | 2.273     | 13277157  | 769559   | 2.804  | 3.811    |
| 11    | 2.809     | 9929799   | 616460   | 2.097  | 3.053    |
| 12    | 3.204     | 1387416   | 139016   | 0.293  | 0.688    |
| 13    | 3.521     | 1305201   | 95237    | 0.276  | 0.472    |
| 14    | 3.744     | 946874    | 59174    | 0.200  | 0.293    |
| 15    | 4.414     | 1095892   | 46641    | 0.231  | 0.231    |
| 16    | 4.760     | 453410    | 43370    | 0.096  | 0.215    |
| 17    | 5.041     | 1411520   | 145617   | 0.298  | 0.721    |
| 18    | 5.280     | 1949047   | 147338   | 0.412  | 0.730    |
| 19    | 5.787     | 5224      | 750      | 0.001  | 0.004    |
| 20    | 6.095     | 451665    | 22417    | 0.095  | 0.111    |
| 21    | 6.656     | 1017814   | 58825    | 0.215  | 0.291    |
| 22    | 7.781     | 571539    | 20990    | 0.121  | 0.104    |
| 23    | 8.768     | 5317987   | 330159   | 1.123  | 1.635    |
| 24    | 9.375     | 110921403 | 6602710  | 23.428 | 32.700   |
| 25    | 11.453    | 124597    | 5373     | 0.026  | 0.027    |
| 26    | 12.083    | 2085      | 178      | 0.000  | 0.001    |
| 27    | 13.108    | 307339272 | 10240837 | 64.914 | 50.717   |
| 28    | 13.688    | 2288397   | 147591   | 0.483  | 0.731    |
| 29    | 14.100    | 6529740   | 355492   | 1.379  | 1.761    |
| 30    | 15.673    | 58370     | 2410     | 0.012  | 0.012    |
| 31    | 16.928    | 1103      | 341      | 0.000  | 0.002    |
| 32    | 16.996    | 2634      | 339      | 0.001  | 0.002    |
| 33    | 17.163    | 1982      | 259      | 0.000  | 0.001    |

| Peak# | Ret. Time | Area      | Height   | Area %  | Height % |
|-------|-----------|-----------|----------|---------|----------|
| 34    | 17.659    | 158681    | 6498     | 0.034   | 0.032    |
| 35    | 18.209    | 7929      | 1618     | 0.002   | 0.008    |
| 36    | 18.268    | 15465     | 1522     | 0.003   | 0.008    |
| 37    | 19.225    | 1867656   | 66718    | 0.394   | 0.330    |
| 38    | 20.236    | 4000676   | 127834   | 0.845   | 0.633    |
| 39    | 21.350    | 3695      | 1071     | 0.001   | 0.005    |
| 40    | 21.436    | 6687      | 884      | 0.001   | 0.004    |
| 41    | 21.613    | 2794      | 524      | 0.001   | 0.003    |
| 42    | 21.694    | 2127      | 496      | 0.000   | 0.002    |
| 43    | 21.808    | 3639      | 780      | 0.001   | 0.004    |
| 44    | 22.206    | 95013     | 3134     | 0.020   | 0.016    |
| 45    | 22.720    | 8402      | 1045     | 0.002   | 0.005    |
| 46    | 22.883    | 8293      | 1080     | 0.002   | 0.005    |
| 47    | 22.994    | 16891     | 1144     | 0.004   | 0.006    |
| 48    | 23.560    | 1094      | 360      | 0.000   | 0.002    |
| 49    | 23.923    | 38259     | 2737     | 0.008   | 0.014    |
| 50    | 24.001    | 25673     | 2705     | 0.005   | 0.013    |
| 51    | 24.165    | 18076     | 2178     | 0.004   | 0.011    |
| 52    | 24.343    | 19956     | 1742     | 0.004   | 0.009    |
| Total |           | 473456802 | 20191931 | 100.000 | 100.000  |

Day 1 unknown sample+dihydrocapsaicin (50ppm)



| Peak# | Ret. Time | Area      | Height   | Area % | Height % |
|-------|-----------|-----------|----------|--------|----------|
| 1     | 0.274     | 1773      | 272      | 0.000  | 0.001    |
| 2     | 0.363     | 1661      | 455      | 0.000  | 0.002    |
| 3     | 0.609     | 3112      | 478      | 0.001  | 0.002    |
| 4     | 0.772     | 2854      | 451      | 0.001  | 0.002    |
| 5     | 0.922     | 2162      | 638      | 0.000  | 0.003    |
| 6     | 0.979     | 1958      | 555      | 0.000  | 0.002    |
| 7     | 1.066     | 2841      | 631      | 0.001  | 0.003    |
| 8     | 1.137     | 2314      | 624      | 0.000  | 0.003    |
| 9     | 1.281     | 5735      | 729      | 0.001  | 0.003    |
| 10    | 1.381     | 4208      | 672      | 0.001  | 0.003    |
| 11    | 1.728     | 1084095   | 167781   | 0.199  | 0.672    |
| 12    | 2.281     | 19132084  | 1045029  | 3.509  | 4.188    |
| 13    | 2.811     | 11684554  | 881704   | 2.143  | 3.534    |
| 14    | 3.048     | 5389656   | 305739   | 0.989  | 1.225    |
| 15    | 3.522     | 2104030   | 149733   | 0.386  | 0.600    |
| 16    | 3.769     | 1526828   | 103471   | 0.280  | 0.415    |
| 17    | 4.405     | 1330923   | 53204    | 0.244  | 0.213    |
| 18    | 4.756     | 826693    | 79888    | 0.152  | 0.320    |
| 19    | 5.036     | 1656843   | 172993   | 0.304  | 0.693    |
| 20    | 5.273     | 2579592   | 199730   | 0.473  | 0.800    |
| 21    | 5.767     | 2204      | 463      | 0.000  | 0.002    |
| 22    | 6.082     | 642070    | 40499    | 0.118  | 0.162    |
| 23    | 6.648     | 1167213   | 68659    | 0.214  | 0.275    |
| 24    | 7.780     | 582585    | 28206    | 0.107  | 0.113    |
| 25    | 8.764     | 8532903   | 547076   | 1.565  | 2.193    |
| 26    | 9.400     | 181609694 | 10231637 | 33.312 | 41.006   |
| 27    | 11.100    | 2416      | 497      | 0.000  | 0.002    |
| 28    | 11.379    | 103143    | 4136     | 0.019  | 0.017    |
| 29    | 11.924    | 5752      | 386      | 0.001  | 0.002    |
| 30    | 12.341    | 2617      | 279      | 0.000  | 0.001    |
| 31    | 12.858    | 288806931 | 10179190 | 52.975 | 40.796   |
| 32    | 13.683    | 1139119   | 104895   | 0.209  | 0.420    |
| 33    | 14.084    | 4312073   | 265897   | 0.791  | 1.066    |

| Peak# | Ret. Time | Area      | Height   | Area %  | Height % |
|-------|-----------|-----------|----------|---------|----------|
| 34    | 17.657    | 1503153   | 24186    | 0.276   | 0.097    |
| 35    | 18.633    | 8886      | 8894     | 0.002   | 0.036    |
| 36    | 19.214    | 2572875   | 81374    | 0.472   | 0.326    |
| 37    | 20.223    | 6589811   | 179821   | 1.209   | 0.721    |
| 38    | 21.467    | 1122      | 286      | 0.000   | 0.001    |
| 39    | 21.846    | 1216      | 337      | 0.000   | 0.001    |
| 40    | 22.204    | 41177     | 3526     | 0.008   | 0.014    |
| 41    | 22.267    | 56236     | 3549     | 0.010   | 0.014    |
| 42    | 22.683    | 2921      | 734      | 0.001   | 0.003    |
| 43    | 22.763    | 2396      | 673      | 0.000   | 0.003    |
| 44    | 22.828    | 3234      | 647      | 0.001   | 0.003    |
| 45    | 23.001    | 4260      | 436      | 0.001   | 0.002    |
| 46    | 23.790    | 27823     | 2491     | 0.005   | 0.010    |
| 47    | 23.907    | 48833     | 3155     | 0.009   | 0.013    |
| 48    | 24.124    | 44151     | 2610     | 0.008   | 0.010    |
| 49    | 24.478    | 10609     | 1332     | 0.002   | 0.005    |
| 50    | 24.652    | 2231      | 540      | 0.000   | 0.002    |
| 51    | 24.791    | 2575      | 309      | 0.000   | 0.001    |
| Total |           | 545176145 | 24951497 | 100.000 | 100.000  |

Day 2 unknown sample+dihydrocapsaicin (50ppm)



| Peak# | Ret. Time | Area      | Height   | Area % | Height % |
|-------|-----------|-----------|----------|--------|----------|
| 1     | 0.079     | 1311      | 374      | 0.000  | 0.001    |
| 2     | 0.275     | 5723      | 693      | 0.001  | 0.003    |
| 3     | 0.385     | 1574      | 422      | 0.000  | 0.002    |
| 4     | 0.511     | 6841      | 746      | 0.001  | 0.003    |
| 5     | 0.883     | 1511      | 409      | 0.000  | 0.002    |
| 6     | 1.397     | 1181      | 263      | 0.000  | 0.001    |
| 7     | 1.725     | 1382387   | 217928   | 0.224  | 0.826    |
| 8     | 2.292     | 22970846  | 1222304  | 3.716  | 4.634    |
| 9     | 2.812     | 14675001  | 1116492  | 2.374  | 4.233    |
| 10    | 3.056     | 4338176   | 428490   | 0.702  | 1.625    |
| 11    | 3.222     | 4088951   | 349118   | 0.662  | 1.324    |
| 12    | 3.508     | 1961567   | 184183   | 0.317  | 0.698    |
| 13    | 3.764     | 2120564   | 140697   | 0.343  | 0.533    |
| 14    | 4.161     | 474636    | 47377    | 0.077  | 0.180    |
| 15    | 4.390     | 1006010   | 53246    | 0.163  | 0.202    |
| 16    | 4.755     | 1196238   | 116201   | 0.194  | 0.441    |
| 17    | 5.034     | 1837858   | 188581   | 0.297  | 0.715    |
| 18    | 5.270     | 3013651   | 232760   | 0.488  | 0.883    |
| 19    | 6.079     | 950162    | 61694    | 0.154  | 0.234    |
| 20    | 6.649     | 1467451   | 78453    | 0.237  | 0.297    |
| 21    | 7.784     | 760483    | 41036    | 0.123  | 0.156    |
| 22    | 8.763     | 10768975  | 689185   | 1.742  | 2.613    |
| 23    | 9.458     | 221497790 | 10232919 | 35.836 | 38.798   |
| 24    | 11.246    | 43129     | 1588     | 0.007  | 0.006    |
| 25    | 11.858    | 1387      | 403      | 0.000  | 0.002    |
| 26    | 11.973    | 2174      | 350      | 0.000  | 0.001    |
| 27    | 12.348    | 4010      | 474      | 0.001  | 0.002    |
| 28    | 12.842    | 307514224 | 10204799 | 49.753 | 38.692   |
| 29    | 13.683    | 1375168   | 125064   | 0.222  | 0.474    |
| 30    | 14.076    | 4860270   | 297365   | 0.786  | 1.127    |
| 31    | 15.459    | 1275      | 448      | 0.000  | 0.002    |
| 32    | 15.737    | 48774     | 2613     | 0.008  | 0.010    |
| 33    | 17.039    | 2838      | 316      | 0.000  | 0.001    |

| Peak# | Ret. Time | Area      | Height   | Area %  | Height % |
|-------|-----------|-----------|----------|---------|----------|
| 34    | 17.640    | 406358    | 15937    | 0.066   | 0.060    |
| 35    | 18.162    | 67848     | 3875     | 0.011   | 0.015    |
| 36    | 19.211    | 2462574   | 87470    | 0.398   | 0.332    |
| 37    | 20.222    | 6493618   | 208435   | 1.051   | 0.790    |
| 38    | 21.849    | 3712      | 852      | 0.001   | 0.003    |
| 39    | 22.178    | 49261     | 3898     | 0.008   | 0.015    |
| 40    | 22.263    | 64797     | 4106     | 0.010   | 0.016    |
| 41    | 23.055    | 1389      | 269      | 0.000   | 0.001    |
| 42    | 23.854    | 48703     | 3616     | 0.008   | 0.014    |
| 43    | 23.984    | 59174     | 3867     | 0.010   | 0.015    |
| 44    | 24.213    | 22618     | 2609     | 0.004   | 0.010    |
| 45    | 24.371    | 18908     | 1884     | 0.003   | 0.007    |
| 46    | 24.596    | 5771      | 853      | 0.001   | 0.003    |
| Total |           | 618086867 | 26374663 | 100.000 | 100.000  |



Day 3 unknown sample+dihydrocapsaicin (50ppm)

| Peak# | Ret. Time | Area      | Height   | Area % | Height % |
|-------|-----------|-----------|----------|--------|----------|
| 1     | 0.258     | 2534      | 425      | 0.000  | 0.002    |
| 2     | 0.648     | 1514      | 298      | 0.000  | 0.001    |
| 3     | 1.135     | 1027      | 248      | 0.000  | 0.001    |
| 4     | 1.261     | 1216      | 342      | 0.000  | 0.001    |
| 5     | 1.726     | 1483595   | 229814   | 0.243  | 0.879    |
| 6     | 2.296     | 23175432  | 1211381  | 3.801  | 4.631    |
| 7     | 2.811     | 15188962  | 1195180  | 2.491  | 4.569    |
| 8     | 3.056     | 8577678   | 463877   | 1.407  | 1.773    |
| 9     | 3.508     | 1817044   | 173398   | 0.298  | 0.663    |
| 10    | 3.772     | 2285640   | 146952   | 0.375  | 0.562    |
| 11    | 4.160     | 548098    | 51515    | 0.090  | 0.197    |
| 12    | 4.397     | 936505    | 50933    | 0.154  | 0.195    |
| 13    | 4.753     | 1409740   | 136924   | 0.231  | 0.523    |
| 14    | 5.032     | 2198858   | 225608   | 0.361  | 0.863    |
| 15    | 5.267     | 3229017   | 247295   | 0.530  | 0.945    |
| 16    | 6.076     | 1047054   | 67690    | 0.172  | 0.259    |
| 17    | 6.646     | 1489701   | 81282    | 0.244  | 0.311    |
| 18    | 7.781     | 765593    | 40245    | 0.126  | 0.154    |
| 19    | 8.761     | 10514313  | 669986   | 1.724  | 2.561    |
| 20    | 9.458     | 218699687 | 10230729 | 35.867 | 39.114   |
| 21    | 11.156    | 2851      | 1012     | 0.000  | 0.004    |
| 22    | 11.229    | 49842     | 1946     | 0.008  | 0.007    |
| 23    | 12.060    | 2435      | 382      | 0.000  | 0.001    |
| 24    | 12.842    | 301071858 | 10196699 | 49.376 | 38.984   |
| 25    | 13.680    | 1269855   | 116373   | 0.208  | 0.445    |
| 26    | 14.073    | 4494879   | 275591   | 0.737  | 1.054    |
| 27    | 15.740    | 51830     | 4460     | 0.009  | 0.017    |
| 28    | 15.848    | 35867     | 4932     | 0.006  | 0.019    |
| 29    | 15.953    | 27587     | 5136     | 0.005  | 0.020    |
| 30    | 16.034    | 119025    | 5106     | 0.020  | 0.020    |
| 31    | 17.059    | 1877      | 437      | 0.000  | 0.002    |
| 32    | 17.639    | 375792    | 14631    | 0.062  | 0.056    |
| 33    | 18.203    | 48283     | 3201     | 0.008  | 0.012    |

| Peak# | Ret. Time | Area      | Height   | Area %  | Height % |
|-------|-----------|-----------|----------|---------|----------|
| 34    | 19.201    | 2370717   | 83772    | 0.389   | 0.320    |
| 35    | 20.213    | 6221165   | 199542   | 1.020   | 0.763    |
| 36    | 22.198    | 47231     | 3465     | 0.008   | 0.013    |
| 37    | 22.264    | 49723     | 3494     | 0.008   | 0.013    |
| 38    | 22.754    | 1369      | 360      | 0.000   | 0.001    |
| 39    | 23.897    | 62026     | 3568     | 0.010   | 0.014    |
| 40    | 24.009    | 29703     | 3192     | 0.005   | 0.012    |
| 41    | 24.188    | 12940     | 2457     | 0.002   | 0.009    |
| 42    | 24.277    | 30472     | 2372     | 0.005   | 0.009    |
| Total |           | 609750535 | 26156244 | 100.000 | 100.000  |

Day 4 unknown sample+dihydrocapsaicin (50ppm)



| Detector A | A Ch I | 280nm · | - 320nm |
|------------|--------|---------|---------|
|------------|--------|---------|---------|

| Peak# | Ret. Time | Area      | Height   | Area % | Height % |
|-------|-----------|-----------|----------|--------|----------|
| 1     | 0.208     | 2303      | 437      | 0.000  | 0.002    |
| 2     | 0.330     | 1900      | 528      | 0.000  | 0.002    |
| 3     | 0.456     | 4949      | 773      | 0.001  | 0.003    |
| 4     | 0.525     | 3776      | 814      | 0.001  | 0.003    |
| 5     | 0.592     | 2143      | 790      | 0.000  | 0.003    |
| 6     | 0.720     | 6224      | 1091     | 0.001  | 0.004    |
| 7     | 0.788     | 6469      | 1222     | 0.001  | 0.005    |
| 8     | 0.950     | 7082      | 1259     | 0.001  | 0.005    |
| 9     | 0.967     | 5041      | 1252     | 0.001  | 0.005    |
| 10    | 1.048     | 10796     | 1355     | 0.002  | 0.005    |
| 11    | 1.220     | 7390      | 817      | 0.001  | 0.003    |
| 12    | 1.724     | 1286847   | 202017   | 0.219  | 0.786    |
| 13    | 2.295     | 21630384  | 1156354  | 3.685  | 4.500    |
| 14    | 2.810     | 13827616  | 1087565  | 2.356  | 4.232    |
| 15    | 3.054     | 7904184   | 416797   | 1.347  | 1.622    |
| 16    | 3.517     | 1949972   | 168596   | 0.332  | 0.656    |
| 17    | 3.696     | 1719335   | 121738   | 0.293  | 0.474    |
| 18    | 4.163     | 652774    | 46215    | 0.111  | 0.180    |
| 19    | 4.422     | 698084    | 47458    | 0.119  | 0.185    |
| 20    | 4.750     | 1186319   | 115708   | 0.202  | 0.450    |
| 21    | 5.028     | 1832688   | 186114   | 0.312  | 0.724    |
| 22    | 5.265     | 2591594   | 202122   | 0.442  | 0.787    |
| 23    | 6.069     | 825459    | 53101    | 0.141  | 0.207    |
| 24    | 6.639     | 1342080   | 74627    | 0.229  | 0.290    |
| 25    | 7.293     | 2540      | 544      | 0.000  | 0.002    |
| 26    | 7.779     | 672238    | 37102    | 0.115  | 0.144    |
| 27    | 8.759     | 9938876   | 640011   | 1.693  | 2.491    |
| 28    | 9.450     | 211559823 | 10229970 | 36.044 | 39.809   |
| 29    | 11.260    | 55389     | 2199     | 0.009  | 0.009    |
| 30    | 11.978    | 1545      | 362      | 0.000  | 0.001    |
| 31    | 12.346    | 5023      | 430      | 0.001  | 0.002    |
| 32    | 12.850    | 290227515 | 10205821 | 49.447 | 39.715   |
| 33    | 13.675    | 1241153   | 111529   | 0.211  | 0.434    |

| Peak# | Ret. Time | Area      | Height   | Area %  | Height % |
|-------|-----------|-----------|----------|---------|----------|
| 34    | 14.070    | 3696187   | 232339   | 0.630   | 0.904    |
| 35    | 17.634    | 1341499   | 28572    | 0.229   | 0.111    |
| 36    | 18.106    | 413617    | 15391    | 0.070   | 0.060    |
| 37    | 18.625    | 9532      | 9545     | 0.002   | 0.037    |
| 38    | 19.189    | 2704107   | 85531    | 0.461   | 0.333    |
| 39    | 20.205    | 7304141   | 197370   | 1.244   | 0.768    |
| 40    | 22.227    | 119095    | 4144     | 0.020   | 0.016    |
| 41    | 22.685    | 14110     | 1221     | 0.002   | 0.005    |
| 42    | 22.912    | 12366     | 1213     | 0.002   | 0.005    |
| 43    | 23.871    | 112565    | 3437     | 0.019   | 0.013    |
| 44    | 24.485    | 5235      | 781      | 0.001   | 0.003    |
| 45    | 24.633    | 1591      | 507      | 0.000   | 0.002    |
| 46    | 24.747    | 1406      | 237      | 0.000   | 0.001    |
| 47    | 24.824    | 1502      | 458      | 0.000   | 0.002    |
| Total |           | 586946464 | 25697462 | 100.000 | 100.000  |

Day 5 unknown sample+dihydrocapsaicin (50ppm)



| Peak# | Ret. Time | Area      | Height   | Area % | Height % |
|-------|-----------|-----------|----------|--------|----------|
| 1     | 0.208     | 18919     | 1722     | 0.003  | 0.006    |
| 2     | 0.330     | 29891     | 1705     | 0.005  | 0.006    |
| 3     | 1.023     | 1638      | 409      | 0.000  | 0.002    |
| 4     | 1.150     | 2206      | 418      | 0.000  | 0.002    |
| 5     | 1.372     | 8430      | 946      | 0.001  | 0.004    |
| 6     | 1.432     | 2435      | 707      | 0.000  | 0.003    |
| 7     | 1.784     | 1766594   | 249087   | 0.311  | 0.931    |
| 8     | 2.321     | 13129666  | 1029148  | 2.314  | 3.846    |
| 9     | 2.482     | 15626598  | 1114676  | 2.754  | 4.165    |
| 10    | 2.809     | 23150155  | 1891777  | 4.080  | 7.069    |
| 11    | 3.083     | 9715033   | 784981   | 1.712  | 2.933    |
| 12    | 3.617     | 8139803   | 558213   | 1.435  | 2.086    |
| 13    | 3.932     | 4853308   | 343275   | 0.855  | 1.283    |
| 14    | 4.441     | 1335127   | 98889    | 0.235  | 0.370    |
| 15    | 4.747     | 3246755   | 272824   | 0.572  | 1.019    |
| 16    | 5.003     | 574251    | 60210    | 0.101  | 0.225    |
| 17    | 5.266     | 1006289   | 53896    | 0.177  | 0.201    |
| 18    | 5.619     | 92414     | 20594    | 0.016  | 0.077    |
| 19    | 5.991     | 624846    | 27229    | 0.110  | 0.102    |
| 20    | 6.216     | 294945    | 29817    | 0.052  | 0.111    |
| 21    | 6.429     | 579522    | 48473    | 0.102  | 0.181    |
| 22    | 6.773     | 1964046   | 65376    | 0.346  | 0.244    |
| 23    | 7.780     | 982581    | 30934    | 0.173  | 0.116    |
| 24    | 8.758     | 7868807   | 456739   | 1.387  | 1.707    |
| 25    | 9.362     | 145510045 | 8510283  | 25.646 | 31.801   |
| 26    | 10.940    | 3182      | 981      | 0.001  | 0.004    |
| 27    | 11.180    | 48964     | 4293     | 0.009  | 0.016    |
| 28    | 11.286    | 39720     | 5091     | 0.007  | 0.019    |
| 29    | 11.409    | 84546     | 5549     | 0.015  | 0.021    |
| 30    | 13.092    | 308578821 | 10242096 | 54.387 | 38.272   |
| 31    | 13.668    | 2139761   | 138141   | 0.377  | 0.516    |
| 32    | 14.073    | 6174329   | 335061   | 1.088  | 1.252    |
| 33    | 15.688    | 1191      | 286      | 0.000  | 0.001    |

| Peak# | Ret. Time | Area      | Height   | Area %  | Height % |
|-------|-----------|-----------|----------|---------|----------|
| 34    | 15.935    | 10192     | 1531     | 0.002   | 0.006    |
| 35    | 16.049    | 31569     | 2272     | 0.006   | 0.008    |
| 36    | 17.006    | 26986     | 2855     | 0.005   | 0.011    |
| 37    | 17.058    | 22119     | 3077     | 0.004   | 0.011    |
| 38    | 17.633    | 3132289   | 123512   | 0.552   | 0.462    |
| 39    | 18.633    | 19061     | 3496     | 0.003   | 0.013    |
| 40    | 19.182    | 1844372   | 67775    | 0.325   | 0.253    |
| 41    | 20.202    | 4297102   | 133951   | 0.757   | 0.501    |
| 42    | 21.542    | 72548     | 5063     | 0.013   | 0.019    |
| 43    | 21.833    | 5960      | 3022     | 0.001   | 0.011    |
| 44    | 21.915    | 17532     | 3420     | 0.003   | 0.013    |
| 45    | 22.150    | 63537     | 4306     | 0.011   | 0.016    |
| 46    | 22.242    | 77754     | 4308     | 0.014   | 0.016    |
| 47    | 22.714    | 11370     | 1607     | 0.002   | 0.006    |
| 48    | 22.802    | 13664     | 1320     | 0.002   | 0.005    |
| 49    | 23.024    | 11754     | 1212     | 0.002   | 0.005    |
| 50    | 23.175    | 5589      | 1036     | 0.001   | 0.004    |
| 51    | 23.286    | 4285      | 751      | 0.001   | 0.003    |
| 52    | 23.375    | 3184      | 747      | 0.001   | 0.003    |
| 53    | 23.445    | 1418      | 735      | 0.000   | 0.003    |
| 54    | 23.806    | 42851     | 2567     | 0.008   | 0.010    |
| 55    | 23.935    | 11100     | 2322     | 0.002   | 0.009    |
| 56    | 24.006    | 16358     | 2165     | 0.003   | 0.008    |
| 57    | 24.131    | 16442     | 1902     | 0.003   | 0.007    |
| 58    | 24.344    | 13821     | 1431     | 0.002   | 0.005    |
| 59    | 24.496    | 12419     | 947      | 0.002   | 0.004    |
| Total |           | 567380091 | 26761161 | 100.000 | 100.000  |

# **ANOVA RESULTS**

# Moisture content of industrial isot sample

ANOVA

| m.c           |                |    |            |         |      |
|---------------|----------------|----|------------|---------|------|
|               | Sum of Squares | Df | MeanSquare | F       | Sig. |
| BetweenGroups | 9087,158       | 7  | 1298,165   | 625,385 | ,000 |
| WithinGroups  | 16,606         | 8  | 2,076      |         |      |
| Total         | 9103,765       | 15 |            |         |      |

| ANOVA         |                |    |            |         |       |  |  |
|---------------|----------------|----|------------|---------|-------|--|--|
| asta1         |                |    |            |         |       |  |  |
|               | Sum of Squares | Df | MeanSquare | F       | Sig.  |  |  |
| BetweenGroups | 7701,325       | 7  | 1100,189   | 1,896E3 | ,000, |  |  |
| WithinGroups  | 4,643          | 8  | ,580       |         |       |  |  |
| Total         | 7705,968       | 15 |            |         |       |  |  |

ANOVA

| dpph1         |                |    |            |        |      |
|---------------|----------------|----|------------|--------|------|
|               | Sum of Squares | Df | MeanSquare | F      | Sig. |
| BetweenGroups | 1704,076       | 7  | 243,439    | 95,700 | ,000 |
| WithinGroups  | 20,350         | 8  | 2,544      |        |      |
| Total         | 1724,426       | 15 |            |        |      |

| ANOVA         |                |    |            |         |       |  |  |
|---------------|----------------|----|------------|---------|-------|--|--|
| t.p.c         |                |    |            |         |       |  |  |
|               | Sum of Squares | df | MeanSquare | F       | Sig.  |  |  |
| BetweenGroups | 1,146          | 7  | ,164       | 2,052E3 | ,000, |  |  |
| WithinGroups  | ,001           | 8  | ,000,      |         |       |  |  |
| Total         | 1,147          | 15 |            |         |       |  |  |
|               |                |    |            |         |       |  |  |

| ANOVA         |                |    |            |         |       |  |  |  |
|---------------|----------------|----|------------|---------|-------|--|--|--|
| n.e.b         |                |    |            |         |       |  |  |  |
|               | Sum of Squares | df | MeanSquare | F       | Sig.  |  |  |  |
| BetweenGroups | ,939           | 6  | ,156       | 5,477E3 | ,000, |  |  |  |
| WithinGroups  | ,000,          | 7  | ,000,      |         |       |  |  |  |
| Total         | ,939           | 13 |            |         |       |  |  |  |

ANOVA

| t.p.c         |                |    |            |         |       |
|---------------|----------------|----|------------|---------|-------|
|               | Sum of Squares | df | MeanSquare | F       | Sig.  |
| BetweenGroups | 1,146          | 7  | ,164       | 2,052E3 | ,000, |
| WithinGroups  | ,001           | 8  | ,000,      |         |       |
| Total         | 1,147          | 15 |            |         |       |

| ANOVA         |                |    |            |         |      |  |
|---------------|----------------|----|------------|---------|------|--|
| vit.c         |                |    |            |         |      |  |
|               | Sum of Squares | Df | MeanSquare | F       | Sig. |  |
| BetweenGroups | ,040           | 7  | ,006       | 136,968 | ,000 |  |
| WithinGroups  | ,000           | 8  | ,000,      |         |      |  |
| Total         | ,041           | 15 |            |         |      |  |

| acidity       |                |    |            |         |       |
|---------------|----------------|----|------------|---------|-------|
|               | Sum of Squares | Df | MeanSquare | F       | Sig.  |
| BetweenGroups | 253,950        | 7  | 36,279     | 103,633 | ,000, |
| WithinGroups  | 2,801          | 8  | ,350       |         |       |
| Total         | 256,751        | 15 |            |         |       |

| L             |                |    |            |         |      |  |  |
|---------------|----------------|----|------------|---------|------|--|--|
|               | Sum of Squares | df | MeanSquare | F       | Sig. |  |  |
| BetweenGroups | 310,045        | 7  | 44,292     | 147,460 | ,000 |  |  |
| WithinGroups  | 2,403          | 8  | ,300       |         |      |  |  |
| Total         | 312,448        | 15 |            |         |      |  |  |

### ANOVA

| A             |                |    |            |         |       |
|---------------|----------------|----|------------|---------|-------|
|               | Sum of Squares | df | MeanSquare | F       | Sig.  |
| BetweenGroups | 1200,843       | 7  | 171,549    | 328,614 | ,000, |
| WithinGroups  | 4,176          | 8  | ,522       |         |       |
| Total         | 1205,019       | 15 |            |         |       |

ANOVA

| В             |                |    |            |         |      |
|---------------|----------------|----|------------|---------|------|
|               | Sum of Squares | df | MeanSquare | F       | Sig. |
| BetweenGroups | 742,084        | 7  | 106,012    | 124,955 | ,000 |
| WithinGroups  | 6,787          | 8  | ,848       |         |      |
| Total         | 748,871        | 15 |            |         |      |

|         |                     | m.c    | asta1  | dpph1  | t.p.c  | pН                | vit.c | Acidity | L     | а      | b     | n.e.b  |
|---------|---------------------|--------|--------|--------|--------|-------------------|-------|---------|-------|--------|-------|--------|
| m.c     | Pearson Correlation | 1      | -,478  | ,982'' | -,735' | ,651              | ,771' | -,637   | ,694  | ,807'  | ,900" | -,634  |
|         | Sig. (2-tailed)     |        | ,231   | ,000   | ,038   | ,080              | ,025  | ,089    | ,056  | ,015   | ,002  | ,091   |
|         | Ν                   | 8      | 8      | 8      | 8      | 8                 | 8     | 8       | 8     | 8      | 8     | 8      |
| asta1   | Pearson Correlation | -,478  | 1      | -,479  | ,106   | -,933"            | -,669 | ,175    | -,552 | -,815' | -,765 | ,239   |
|         | Sig. (2-tailed)     | ,231   |        | ,229   | ,803   | ,001              | ,070  | ,678    | ,156  | ,014   | ,027  | ,569   |
|         | Ν                   | 8      | 8      | 8      | 8      | 8                 | 8     | 8       | 8     | 8      | 8     | 8      |
| dpph1   | Pearson Correlation | ,982   | -,479  | 1      | -,824  | ,666              | ,860" | - 749   | ,586  | ,852   | ,893" | -,707  |
|         | Sig. (2-tailed)     | ,000   | ,229   |        | ,012   | ,071              | ,006  | ,032    | ,127  | ,007   | ,003  | ,050   |
|         | Ν                   | 8      | 8      | 8      | 8      | 8                 | 8     | 8       | 8     | 8      | 8     | 8      |
| t.p.c   | Pearson Correlation | -,735' | ,106   | -,824  | 1      | -,291             | -,766 | ,846"   | -,143 | -,585  | -,549 | ,876'' |
|         | Sig. (2-tailed)     | ,038   | ,803   | ,012   |        | ,485              | ,027  | ,008    | ,735  | ,128   | ,159  | ,004   |
|         | Ν                   | 8      | 8      | 8      | 8      | 8                 | 8     | 8       | 8     | 8      | 8     | 8      |
| pН      | Pearson Correlation | ,651   | -,933" | ,666   | -,291  | 1                 | ,808' | -,378   | ,619  | ,926'' | ,888" | -,321  |
|         | Sig. (2-tailed)     | ,080   | ,001   | ,071   | ,485   |                   | ,015  | ,356    | ,102  | ,001   | ,003  | ,438   |
|         | Ν                   | 8      | 8      | 8      | 8      | 8                 | 8     | 8       | 8     | 8      | 8     | 8      |
| vit.c   | Pearson Correlation | ,771   | -,669  | ,860   | -,766  | ,808 <sup>°</sup> | 1     | -,810   | ,331  | ,949"  | ,831  | -,678  |
|         | Sig. (2-tailed)     | ,025   | ,070   | ,006   | ,027   | ,015              |       | ,015    | ,423  | ,000   | ,011  | ,065   |
|         | Ν                   | 8      | 8      | 8      | 8      | 8                 | 8     | 8       | 8     | 8      | 8     | 8      |
| Acidity | Pearson Correlation | -,637  | ,175   | -,749  | ,846   | -,378             | -,810 | 1       | ,062  | -,672  | -,493 | ,544   |
|         | Sig. (2-tailed)     | ,089   | ,678   | ,032   | ,008   | ,356              | ,015  |         | ,884  | ,068   | ,214  | ,164   |
|         | Ν                   | 8      | 8      | 8      | 8      | 8                 | 8     | 8       | 8     | 8      | 8     | 8      |
| L       | Pearson Correlation | ,694   | -,552  | ,586   | -,143  | ,619              | ,331  | ,062    | 1     | ,528   | ,793' | -,253  |
|         | Sig. (2-tailed)     | ,056   | ,156   | ,127   | ,735   | ,102              | ,423  | ,884    |       | ,178   | ,019  | ,546   |
|         | Ν                   | 8      | 8      | 8      | 8      | 8                 | 8     | 8       | 8     | 8      | 8     | 8      |
| а       | Pearson Correlation | ,807'  | -,815' | ,852'' | -,585  | ,926''            | ,949" | -,672   | ,528  | 1      | ,929" | -,507  |
|         | Sig. (2-tailed)     | ,015   | ,014   | ,007   | ,128   | ,001              | ,000  | ,068    | ,178  |        | ,001  | ,199   |
|         | Ν                   | 8      | 8      | 8      | 8      | 8                 | 8     | 8       | 8     | 8      | 8     | 8      |
| b       | Pearson Correlation | ,900"  | -,765  | ,893   | -,549  | ,888"             | ,831  | -,493   | ,793  | ,929"  | 1     | -,537  |
|         | Sig. (2-tailed)     | ,002   | ,027   | ,003   | ,159   | ,003              | ,011  | ,214    | ,019  | ,001   |       | ,170   |
|         | Ν                   | 8      | 8      | 8      | 8      | 8                 | 8     | 8       | 8     | 8      | 8     | 8      |
| n.e.b   | Pearson Correlation | -,634  | ,239   | -,707  | ,876'' | -,321             | -,678 | ,544    | -,253 | -,507  | -,537 | 1      |
|         | Sig. (2-tailed)     | ,091   | ,569   | ,050   | ,004   | ,438              | ,065  | ,164    | ,546  | ,199   | ,170  |        |
|         | Ν                   | 8      | 8      | 8      | 8      | 8                 | 8     | 8       | 8     | 8      | 8     | 8      |

Correlations

\*\*. Correlation is significant at the 0.01 level (2-tailed).

\*. Correlation is significant at the 0.05 level (2-tailed).