CORVINUS
UNIVERSITY of
BUDAPEST

Faculty of Food Science

INVESTIGATION OF COLOUR AND AROMA PROPERTIES OF RED PEPPER POWDERS

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Doctoral theses

Budapest

2014

PhD School

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Field: Food Science

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The applicant met the requirement of the PhD regulations of the Corvinus University of Budapest and the thesis is accepted for the defence process.

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1. INTRODUCTION

Red pepper (*Capsicum annuum* L.) is perhaps the most important spice plant of our country, which has been contributed significantly to establish the reputation of Hungarian cuisine. In its ground form, paprika is used in most of the traditional Hungarian meals and it is among the ingredients of numerous canned and meat products. Hungary is considered worldwide as a recognized grower of this spice, and red pepper became a *Hungaric* (*i.e.* gastronomic speciality), it is a well-known and popular commodity in abroad as well. The members of *Capsicum* genus are applied generally in many other countries of the world owing to their bright colour, pleasant aroma and pungent taste of certain varieties'.

In the last two decades, the crop land, yield and produced red pepper powder amount has been decreased continuously also in Hungary and in Europe. In parallel with this process, inexpensive import paprika from South America, South Africa and Asia is flowing to the European markets. Blending them with Hungarian powder red pepper products at more favourable price can be produced. In terms of the cost of production, Hungarian red pepper will never be competitive with those deriving from countries with warmer climate. Consequently, the future of the cultivation of this spice plant can be insurable with the manufacture of products with special quality (for example protected designation of origin). These kinds of products inspire more confidence in export markets and can be sold at higher price.

The aroma and colour of red pepper are important quality parameters and essential standards of products prepared from it. These organoleptic properties play important role in the choice of consumers and can give a useful help in determining the ripening stage of the plant, during breeding and in identification of variety and provenance (*i.e.* verification of origin) as well. Blending different import peppers with Hungarian ones is increasingly widespread, in order to improve colour properties. Identification of characteristic aroma constituents referring provenance clearly indicates the origin diverse from Hungary, while individual odorants typical of Hungarian varieties can verify domestic raw material.

2. OBJECTIVES

The aim of my experimental work was the investigation of volatile constituents in red pepper powders with GC-MS method. With the examination of aroma properties, important information can be obtained regarding the origin, ripening state and in generally, the quality of the raw material.

Following the selection of proper sampling method, the determination of fragrance composition of red pepper varieties from Szeged growing region, and aroma changes resulting during storage at different temperatures were among my purposes. Namely, storage conditions influence significantly the organoleptic properties of red pepper and powder.

Besides domestic varieties, ground paprika samples and products from abroad were examined. The results of some Hungarian varieties and peppers cultivated in other countries were compared to identify individual aroma compounds clearly indicating foreign provenances. Further on, with the examination of a multitude of red pepper powders from different origin the identification (or narrowing – Europe, South America, Asia *etc.*) of the provenance will become possible.

The examinations were expanded also to red pepper powder mixtures. These investigations possess great importance because blending foreign paprika with Hungarian varieties becomes widely distributed to improve colour properties of the products. With the identification of marker constituents characteristic exclusively to Hungarian varieties – or appearing only in foreign powders – the verification of blending raw materials from abroad can be feasible.

In addition to the volatile fraction of red pepper powder, the investigation of other important organoleptic property, its colour was among the objectives of the dissertation. Besides following the effect of storage to colour, the colour improving effect of blending with numerical data were intended to examine. The colour characteristics and pigment contents of red pepper powders from diverse origin were investigated for this purpose.

3. MATERIALS AND METHODS

Examined samples:

For the aroma and colour examinations, the following dried red pepper pericarps and powders were obtained:

- cultivar identical dried red pepper pericarps were acquired by the help of Red Pepper Research-Development P.B.C. from Szeged in 2007. The examined cultivars were as follows: Szegedi 20, Szegedi 80, Remény (sweet varieties), Szegedi 178, Szegedi F-03 (hot varieties).
- Szegedi Különleges powder, and paprika products from significant growing countries (Spain, Turkey and Peru) and blended powders (Hungarian-Peruvian, Hungarian-Spanish, Hungarian-Serbian and Spanish-Serbian) were purchased from trade in 2010.

Examination methods:

In the course of aroma investigations various sampling methods were applied: steam distillation, simultaneous distillation-extraction (SDE) and solid phase microextraction (SPME) with fibres of different polarities (nonpolar, polar and bipolar). The instrumental measurements were conducted with GC-MS. The evaluation of the results was performed with aromaspectra method elaborated at the Department of Food Chemistry and Nutrition.

The colour characteristics of red pepper powders were measured with colorimeter and the results were supplied in CIELAB colour space.

The determination of total pigment content was performed according to the MSZ 9681-5:2002 standard with spectrophotometric method. The results were given as ASTA units in whole numbers.

4. RESULTS

4.1. Results of the aroma examinations

4.1.1. The effect of sampling to the aroma composition

Three different sampling methods were tested to gain as much information as possible regarding the fragrance of red pepper powders. The aroma pictures gained by various extraction methods were divergent from each other, namely the ratio of recovered component groups depends greatly on the applied procedure. The most odour constituents were extracted from the paprika samples by the distillation methods, although one of the SPME fibres (PDMS-DVB) was adsorbed fragrance compounds in great number.

From the aroma compounds appearing in the volatile fraction, odour-active terpene components arising from secondary metabolism processes were the main information carriers regarding the nature of the raw material (species, variety and provenance). In this manner, for species identification and describing cultivar characteristics, distillation methods are the most appropriate for aroma extraction. Terpene constituents could namely be detected in greatest number with these procedures. Among SPME fibres PDMS-DVB proved to be the most effective in this respect. Another compound group with great odour activity was those of heterocyclic constituents' developing during processing. Their appearance on the chromatograms indicates the fact of heat load merely. For their detection, SPME fibres were the most suitable. Alcohols, aldehydes and ketones are generally lipid oxidation products and can designate heat treatment and the ripening stage of the fruit (especially the C_6 compounds). These odour constituents could be adsorbed by the Carboxen-PDMS fibre in greatest number.

In the course of my further experimental work SDE method was applied for the investigation of volatile fraction in diverse red pepper powders from different origin. With this procedure, alterations occurring during cooking can be modelled as well, so an overall picture from volatiles arising both during ripening and processing could be obtained.

4.1.2. Aroma characteristics of domestic varieties

More than 170 aroma constituents were detected in all of the dried red pepper pericarps with simultaneous distillation-extraction (SDE) method, among which 115 were present in all five varieties, although with different ratio. Every examined cultivar possessed particular fragrance pattern, which has been caused by the presence of individual odorants and the diverse ratio of common fragrance materials. Among unique aroma compounds, terpenes deserve special attention since other volatiles (mostly fatty acids, esters, N-heterocycles, alcohols, aldehydes ketones and hydrocarbons) are less odour-active. Most of them form in oxidation reactions during processing and are not suitable for cultivar identification. On the basis of terpene aromaspectra and the peak area share of odour-active volatiles from the total aroma, sweet varieties (Remény, Szegedi 20 and Szegedi 80) seemed more fragrant than hot ones (Szegedi F-03 and Szegedi 178).

4.1.3. The alteration of red pepper aroma during storage

The effect of different temperatures to the aroma composition of red pepper during storage was examined. As a result of diverse temperatures, the number of the identified fragrance constituents has virtually not been changed under storing (cold storage: from 167 to 171 components, ambient storage: from 167 to 161 constituents). Peak areas of volatiles with low boiling points (mostly terpenes, different heterocyclic and benzene-ringed constituents) have been decreased significantly to the end of storing. This alteration was greater at ambient temperature owing to the reaction accelerating and vaporization increasing effects of higher temperature. The production of certain carotenoid degradation products (*β*-ionone, dihydroactinidiolide) and less odour-active fatty acids (lauric, miristic and palmitic acids) and esters (methyl palmitate, linolenic acid methyl and ethyl esters) has been increased during storage, especially at higher temperature.

4.1.4. The fragrance features of foreign red pepper powders

During aroma examinations, red pepper powders and flakes from three significant producing countries: Peru, Turkey and Spain were investigated.

In Peruvian red peppers the most outstanding result was the appearance of some highly intensive, turmeric-characteristic volatiles (turmerone compounds, zingiberene, *β*-sesquiphellandrene, *α*-curcumene, *α*-phellandrene, *α*-atlantone) in sweet paprika powder. The explanation for their presence can be the diverse fragrance composition of the raw material – owing to the remote provenance or cultivar – as colour deepening with curcuma is probably not effective. In Peruvian red pepper powders altogether 18 terpene constituents were identified, which could not be detected in Hungarian varieties – these volatiles therefore can be the markers of foreign origin. All groups of heterocyclic constituents (S-, N- and O-containing odorants) were present in lower ratio as experienced in domestic cultivars. This result was in all probability owing to the lower heat load during processing, which was caused by sun drying in relation to artificial drying applied in Hungary.

Four red pepper samples from Turkey – sweet and hot powders and hot flakes – were also studied. During fragrance investigations ground paprika products proved to be more fragrant than coarse ones. Organoleptic differences were confirmed by instrumental examinations: the main variance seemed to be in the number and intensity of odour-active terpene compounds. The provenance, red pepper cultivars and varieties diverse from Hungarian were verified by the appearance of individual fragrance compounds. Turkish red peppers contained these „marker" odorants in especially great number. The explanation for this result can be the processed species different from *Capsicum annuum* like *C. frutescens* or *C. baccatum*. Some of these characteristic volatiles were: *cis*-dihydrocarvone, *trans*-carveol, *δ*-elemene, *α*-cedrene, *cis*-*α*-bisabolene, *α*-calacorene. Heterocyclic constituents were present in Turkish products in lower ratio than in domestic cultivars – similarly to the Peruvian red peppers – which may be the consequence of milder heat load (sun drying).

In the volatile fractions of Spanish sweet and hot ground red peppers a great number of aroma constituents were detected (more than 150). Fragrance compounds verifying the processing of raw materials divergent from Hungarian were found in these paprika samples as well. Similarly to the other spices these substances were terpenes like *α*-humulene, *β*-cadinene, *δ*-4-carene and valerenol. Every volatiles possessing turmeric character and identified in Peruvian sweet ground paprika also were detected in Spanish sweet powder. This result can be explained by the processing of a characteristic variety not cultivated in Hungary or blending with the mentioned Peruvian cultivar. The low total intensity of fragrances containing heteroatoms in Spanish red peppers is owing to the milder heat load of sun drying. In Spanish hot pepper benzene compounds with roasted, smoky, woody characters referring to the special drying method (smoking) were detected.

4.1.5. The aroma composition of ground red pepper mixtures

The examinations were expanded to blended (Peruvian-Hungarian, Hungarian-Spanish, Hungarian-Serbian and Serbian-Spanish) red pepper powders. On the basis of the chromatograms and the number of detected fragrance compounds Hungarian-Serbian and Serbian-Spanish products were the most fragrant, following Peruvian-Hungarian and Hungarian-Spanish ones. Volatile compounds indicating foreign raw materials were terpenes and benzene-ringed substances. These odorants have been referred to the import origin instead of concrete provenances. In Hungarian-Peruvian product four terpenes were identified as markers of foreign origin, but intensive turmerone compounds of the sweet Peruvian powder

were not among these volatiles. The indicatives of import origin were β-pinene, l-carvone and γ-himachalene in Hungarian-Spanish blend. Contrary to the other mixtures, the markers of Serbian origin were benzene constituents like 4-allylphenol and myristicine.

In Hungarian cultivars from Szeged growing region six common terpene compounds were identified which were not present in red pepper products from abroad (4-ethyl-2,6-xylenol, α-muurolene, p-mentha-3,8-diene, elemol, liguhodgsonal, 3,4-dimethoxy-bicyclo[4.2.0]octa-1,3,5-trien-7-ol). These odorants were in search to identify the process of Hungarian raw material during blending. Only two volatiles (4-ethyl-2,6-xylenol and liguhodgsonal) were managed to detect in each products, so the verification of Hungarian origin was only partially successful. Consequently, the detection of import origin in blended products seemed more reliable than verification of partly domestic source.

4.1.6. Comparison of fragrance features of pungent and sweet ground red peppers

Although capsaicinoids triggering hot taste of red peppers do not appear in the extracts owing to their low volatilities, characteristic odorants indicating the pungency of raw material were detected. These volatiles were present exclusively in hot varieties, irrespective of their provenance. These aroma constituents were sesquiterpenes of himachalane and longipinane skeletons: α-, β-, γ-himachalene, longicyclene and longifolene. α-himachalene was only part of Hungarian varieties, while γ-himachalene was detected in foreign products. The presence of this latter odorant can therefore indicate the processing of hot raw material from abroad, as soon as it has been appeared in the volatile fraction of Hungarian-Spanish ground product. The mentioned sesquiterpenes have not been identified in the sweet varieties and ground products, so they can be regarded as the markers of pungency indeed.

4.2. The results of colour examinations

4.2.1. The colour properties of ground products

According to their colour characteristics, domestic and foreign products were separated from each other. This difference has been appeared principally in the formation of red-green colour coordinate and lightness: both values were higher in domestic cultivars than in red peppers from abroad. That an explicit difference was not observable among domestic and foreign products in case of yellow-blue coordinate. On the basis of their colour characteristics, blended products were among Hungarian and foreign red peppers: the values of all three characteristics were found between the values on the subject. Their lightness values have been found below those of Hungarians', so these products possessed darker shade. This can be the main reason of blending domestic paprika with those from abroad: deepening of colour.

Similar results were gained with the examination of total pigment content: the ASTA values of blended products proved to be higher than the data concerning Hungarian varieties. Among foreign red pepper samples, the pigment content of Peruvian and Turkish products has been exceeded those of the Hungarian possessing the highest values. The efficiency of ground pepper blending for colour improving can be verified with the measurement of total pigment content as well.

4.2.2. Colour alterations during storage

The formation of colour characteristics and total pigment content were followed during storage of dried red pepper pericarp from Szeged at different temperatures. The colour of red pepper stored at higher temperature became brighter to the end of storing, and the values of red and yellow coordinates showed decrease (a^*) and increase (b^*), this change was more intense at higher temperature. The alteration of lightness proved to be the greatest after 12 month of storage at both temperatures so this colour characteristic can be regarded as the most heat-sensitive.

The decrease of total pigment content was also greater in case of red pepper stored at ambient temperature: after 12 months this reduction was 80 ASTA values (50,6%), while at the temperature of the refrigerator it was only 26 ASTA (16,5 %). This difference can be attributed clearly to the various storage temperatures. The extent of diminution at ambient temperature has been exceeded those of maximally permitted in the concerning decree.

4.3. Novel scientific results

The novel scientific results of the dissertation can be summarized as follows:

1. Owing to the fragrance pattern-determining property of sampling method, prior to the measurement selection have to be made as a function of analytical objective; in case of cultivar/provenance and organoleptic quality identification Likens-Nickerson SDE (perhaps steam distillation, SD); in other cases SPME fibre being suitable for the task. The artefact-forming effect of distillation methods (LN-SDE, SD) have been refuted clearly by the SPME measurements.

2. Two marker constituents of the Hungarian varieties (4-ethyl-2,6-xylenol and liguhodgsonal) found in none of the foreign cultivars have been identified. In domestic red peppers, the number and ratio of odour-active terpene and pyrazine compounds were significantly higher than in foreign and blended products.

3. During storage, the intensities of odorants with lower boiling points have been decreased essentially, while the amount of fatty acids and carotenoid degradation products have been increased significantly – this change proved to be more intensive at higher temperatures in both cases.

4. Every foreign ground product possessed individual fragrance pattern, and characteristic aroma constituents not detected in domestic cultivars were identified in all of these samples. The appearance of these volatiles in Hungarian red pepper powders indicates the process of raw materials deriving from abroad.

5. Independently of provenance, odorants being present exclusively in the volatile fractions of pungent varieties were identified. Beyond the presence of capsaicinoid components, hot cultivars can thus be characterized with aroma constituents of himachalane and longipinane skeletons.

6. According to their colour characteristics, domestic and foreign products have been definitely separated from each other. On the basis of these characteristics, mixed products were among the mentioned groups, so the colour improving effect of blending can be verified with instrumental colour measurements as well. The examination of total pigment content has been verified the success of ground pepper blending, as the pigment content of the mixtures showed more favourable values.

5. CONCLUSIONS AND SUGGESTIONS

As Hungary is considered worldwide as a recognized grower of red pepper and this spice is recorded as a *Hungaric* (*i.e.* gastronomic speciality), appropriate qualifying methods can be possessed to improve its competitiveness and preserve its reputation. The quality and seasoning value of red pepper is determined mostly by its fragrance *i.e.* aroma wealth, so it is especially important to occupy reliable procedures that can be applied routinely. My results can usefully complete international examinations with the fragrance features of several Hungarian red pepper varieties.

With the sampling and evaluation methods introduced in the dissertation, the identification of characteristic aroma compounds indicating the foreign origin of the product became possible. Continuing and expanding the investigations to further domestic and foreign cultivars and varieties, more exact identification of provenance and revealing of differences among various *Capsicum* species can be realizable. During further examinations, the divergences between the aroma compositions of cultivars from two main domestic provenances can be discovered. The detection of characteristic sesquiterpenes of himachalane skeleton in pungent varieties refers unanimously to the presence of capsaicinoid compounds, so with the investigation of fragrance composition some information about pungency can be gained at the same time.

Further objects of the examinations can be the study of aroma composition during the whole life cycle of the plant. In its original habitat, red pepper is namely a perennial spice plant, so in Hungary always the harvest from the first year is consumed. These investigations might show the age in what the plant can yield the fruits of best quality.

Methods of instrumental aroma examinations discussed in the dissertation can be utilized during the breeding process, because its results could help to select *Capsicum* species rich in aroma compounds. In parallel with aroma examinations, the determination of colour characteristics and total pigment content can contribute to the improvement of organoleptic properties of red pepper powders. Instrumental examination of colour improving effect of blending can further help to determine the optimal blending ratio of different paprika varieties. In this manner, regarding the most important characteristics, organoleptic properties can be completed with instrumental control.

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