Quality properties of caraway seed from various sources

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Seeds of caraway *(Carum carvi)* serve as a spice in many foods such as bread, cheese and sauerkraut. Moreover, caraway seed has been used as a medicine. Consenquently, it is on sale not only in food shops but also in health food stores and drug stores. The plant is cultivated in Finland to some extent, but also grows wild. It has been claimed that Finnish caraway seed has a stronger aroma than that grown elsewhere (1). According to Hegi (2), the volatile oil content is higher in wild caraway seed than in seed from cultured plants. If this is true, increasing collection and culture here would be worth while.

The properties that are important in caraway seed have yet to be established as for all the spices and health food herbs basic data for the evaluation of quality are lacking. In our laboratory a wide project is in progress concerning the quality criteria for health food herbs, of which this study of caraway seed forms a part. In this preliminary study some methods of quality evaluation have been applied to a selection of caraway seed samples from various sources to obtain a basis for quality requirements. Interest was focused primarily on the aroma. A further purpose of the study was to obtain information about the aroma of home-grown cultivated and wild caraway seed as a possible basis for increasing culture.

Material

Commercial samples were obtained from food and health food stores and from wholesalers; one sample was from industry. Another sample was purchased from Switzerland. Seeds of wild caraway were collected in southern Finland (3 samples). In all, 4 commercial samples were of foreign origin, and 4 commercial samples of Finnish origin; of the last-mentioned 2 were known to be aged. The list of the samples is as follows:

Samples of foreign origin

I from Switzerland, health food store
II health food store, packed in Germany
III health food store, packed in Germany
IV from Poland, foods wholesale store

Finnish origin, cultivated

V industrial sample, aged (>1 year old)
VI growers sample, aged (>1 year old)

VII growers sample, fresh
VIII food store sample, fresh

Finnish origin, wild

IX collected 6. 8. 1980
X collected 12. 8. 1980
XI collected 12. 8. 1980

Methods of investigation

To evaluate the general quality a preliminary inspection was performed to check the appearance and the purity. At the same time a rough evaluation was made of the odour and taste. Impurities were measured by selection and weighing. The size of the seeds was measured by weighing 1000 seeds.

In the laboratory the weight loss (moisture) was measured by drying the samples to constant weight at 105 °C. Results are given as percentages. Ash was obtained by incineration of ground samples at 550 °C for 2 h (3), and acid-insoluble ash (sand) according to the AOAC method (4).

Volatile oil was assayed by steam distillation (5). Approximately 30 g of caraway seed was ground rapidly in a Bamix spice mill. For each sample assays were done in duplicate. After distillation for 4 h, the volume was read on the scale to the nearest 0.01 ml. The results are given in ml/100 g. The composition of the volatile oil was studied by gas chromatography. The distillate was diluted to 1 : 100 and as an internal standard pentadecane was added. The gas chromatograph was a Carlo Erba Fractovap 2350 combined with flame ionization detector (FID). The column was an FFAP glass capillary column, 30 m, i. d. 0.3 mm. The temperature programming was between 60 and 220 °C, 6° C/min, the temperature of the injector and detector being 230°C. For quantification of the compounds an integrator Varian C DS 111 was combined with the gas chromatograph. These runs gave the contents of carvone and limonene in the oil.

The methods used for sensory measurement of odour threshold values and for characterization of the aroma profile are explained in connection with the results obtained.

Results

Preliminary inspection of the appearance, odour and size of the seeds

In Finland no quality standards have been set for caraway seed. However, according to the Finnish special literature the amount of caraway pedicels or stem particles should not exceed 5% (6). In the literature there are very few standards for caraway seed. Pearson (7) mentions that foreign organic impurities should not exceed 2%, and seeds of other plants are not allowed. In the United States Pharmacopoeia the maximal amount of other seeds and other organic impurities permitted is 3% (8). In the Hungarian standard for spice caraway (9a) the purity shall be 97.5% in commercial and 96.5% in industrial quality, and maximal amounts of defective seeds are 1.5% and 2%, of organic foreign matter 0.8% and 1.2%, and of inorganic foreign matter 0.2% and 0.3%, respectively. The corresponding standard for medicinal caraway seed (9b) allows maximally 3% of stem parts or similar

impurities derived from caraway in class I, 4% in class II, and parts of other plants maximally 1% in class I, 2% in class II.

The preliminary inspection was performed in such a way that samples of the seeds were spread on trays and inspected visually with the naked eye without optical magnification. A check was made for the presence of impurities, such as pedicels or stem parts of caraway or other seeds, and for the presence of dark, extracted seeds, plant diseases, insect damage, moulds, etc., and soil impurities. The size and evenness of the sample were also considered. A preliminary evaluation of the aroma (strength, possible deterioration with age) was also performed. The presence of impurities and any weakening of the aroma were scored separately on a scale of points (negative values) agreed upon in advance as follows:

No defects	0 points
Slightly defective	−1 points
Highly defective (not marketable)	−2 points

The 4–6 panelists who made the inspection were persons already trained in sensory evaluation.

Table 1 gives the results obtained for the impurities and aroma defects and the weights of 1000 seeds.

Table 1.

Impurities, aroma defects, and weights of 1000 seeds in the caraway seed samples

Sample	Impurities points 0–2	Aroma defects points 0–2	Weight of 1000 seeds g
Foreign origin			
I	0.58	0.50	2.671
II	0.08	0.13	2.119
III	0.46	0.13	3.079
IV	1.25	0.38	2.459
Finnish origin, cultivated			
V	1.75	0.88	3.649
VI	0	2	2.896
VII	0.50	0.33	3.880
VIII	0.46	0.38	3.117
Finnish origin, wild			
IX	0.50	0	1.749
X	1.04	0.04	
XI	0.58	0.08	1.912

In sample V, which contained the highest amount of organic impurities, these were weighed separately and found to be 5.6%. In all the other samples the amounts were much smaller. Sample V was industrial raw material, used for extraction of caraway oil, and so the significance of impurities was not great.

The aroma seemed to be weakest in samples V and VI, which were known to be aged.

The size of the seeds was clearly smallest in the samples from wild plants of Finnish origin (see also 10). This feature may be associated with a strong aroma.

Chemical analyses

In the literature, the maximum limit for seed moisture has been set at 15% (11); the Hungarian standards give the value 13% (9a, 9b). Ash content maximally may be 9% (11); Finnish special literature (6) gives the limit 8%. Hungarian standard (9a) sets the limit at 7% for commercial and 8% for industrial quality; standard for medicinal caraway seed (9b) allows 8% in class I and 9% in class II.

For the soil content (acid-insoluble ash) a limit of 2% has been given (11,6), The United States Pharmacopoeia sets the limit at 1.5% (8); the Hungarian standard for spice caraway (9a) sets the limit at 1.2% for commercial and at 2.0% for industrial quality, the standard for medicinal caraway seed (9b) at 1.5% for class I and at 2% for class II. The amount of volatile oil varies between 3 and 7% (12, 13, 14, 11, 17). The requirement in the Hungarian standard for spice caraway (9a) is at least 3% in commercial and 2.8% in industrial quality, and in the standard for medicinal caraway seed (9b) at least 2.5% in class I. The main compounds of the volatile oil are carvone and limonene, which together make up more than 95% of the volatile oil. In good quality caraway seed the proportion of carvone should be high (13, 15).

The following table gives the analytical results and the calculated ratio of carvone to limonene.

Table 2

Weight loss (moisture), ash, acid-insoluble ash, volatile oil, carvone percentage in oil and carvone limonene proportion of the caraway seeds

Sample	Weight loss %	Ash %	Acid insoluble ash, %	Volatile oil ml/100 g	Carvone in oil %	Carvone/ limonene ratio
Foreign origin						
I	5.8	5.8	0.13	4.23	53.6	1.19
II	6.8	5.2	0.04	3.27	51.9	1.11
III	7.0	5.1	0.12	3.11	46.8	0.90
IV	8.3	5.9	0.07	3.81	53.6	1.19
Finnish origin, cultivated						
V	6.0	5.3	0.24	3.48	54.3	1.22
VI	6.5	5.7	0.03	4.90	52.4	1.13
VII	7.3	5.4	<0.01	4.04	53.6	1.19
VIII	9.6	5.4	<0.01	4.13	54.3	1.22
Finnish origin, wild						
IX	6.4	5.4	<0.01	8.03	48.3	0.96
X	6.0	5.6	0.01	7.43	5.33	1.17
XI	5.8	4.9	0.12	6.72	51.5	1.09

The levels of weight loss, ash and acid-insoluble ash were definitely below the limits. The differences between the samples were relatively small. In sample V, where the amount of organic impurities was highest, the acid-insoluble ash was also higher than in the other samples, but even here the value was far below the limits set.

The volatile oil content was clearly higher in the wild-growing Finnish seeds than in cultured Finnish or foreign seeds. In samples V and VI, which were known

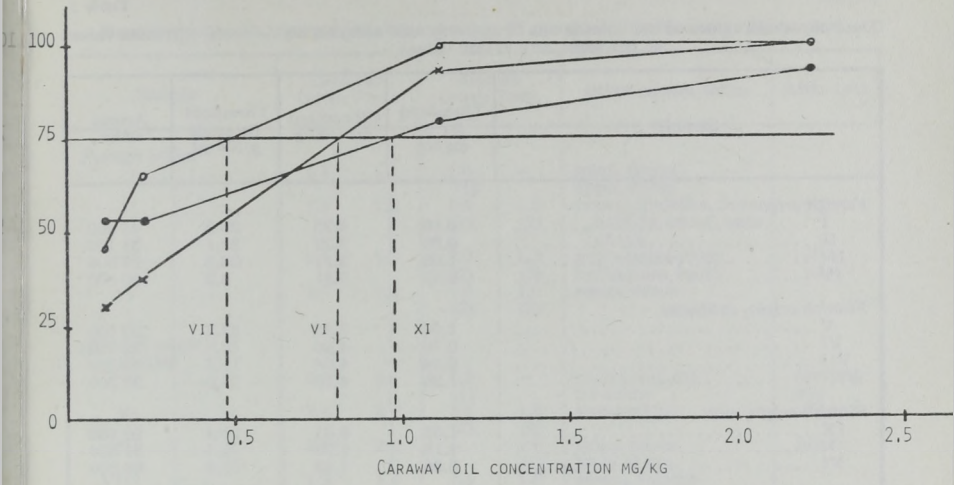

Fig. 1.
Determination of the odour detection threshold values for three of the seed oil samples (VI, VII and XI)

to be aged, the amount of the volatile oil was not clearly lower than in the fresh samples VII and VIII. The carvon content varied relatively little, the mean value being 52.1%; the wild caraway had the lowest values. In the gas chromatographic picture the samples did not vary much. The amounts of compounds present in small quantities, such as α-pinene, β-pinene, myrcene, p-cymene, carveol and dihydrocarveol, were approximately equal in the different samples.

Sensory measurement of the aroma strength by odour threshold assessment

To quantify the aroma of the caraway seed the volatile oils were distilled off and their odour detection thresholds determined. For this purpose, 0.5 ml of each volatile oil was dissolved in 3 ml of ethanol and the solution was made up to 200 ml with water. The water had been destilled before use and treated with active carbon. From the above-mentioned basic solutions further dilutions were prepared for the assessment of the odour thresholds. Four dilutions were used, both weaker and stronger than the detection threshold concentration. For the assessment of the treshold value the duo-trio test was used as described by Schwimmer and Guadagni (16). The threshold value was taken as that concentration at which 75% of the panelists distinguished the sample from plain water (Fig. 1) (17). The assessment was performed at 21 ± 1°C. Samples (200 ml) were presented in 50 ml beakers covered with watch-glasses. The tests were performed in a room planned for the purpose, with individual booths and good ventilation. The panelists were 12–14 trained tasters all accustomed to sensory testing.

From the odour thresholds obtained for the volatile oils the threshold values were calculated for the original caraway seed samples. In addition the aroma

Table 3.

Odour threshold values of the volatile oils of caraway seed samples, oil contents, threshold values of the seeds, and aroma values

Sample	Threshold for oil mg/kg	Oil content ml /100 g	Threshold for seeds g/10^6 ml	Aroma value
Foreign origin				
I	0.89	4.23	23.0	43 200
II	0.96	3.27	32.1	31 100
III	1.03	3.11	36.2	27 500
IV	0.32	3.81	9.2	100 800
Finnish origin, cultivated				
V	1.03	3.48	32.3	30 700
VI	0.76	4.90	16.8	58 700
VII	0.44	4.04	11.8	84 200
VIII	1.25	4.16	33.0	30 300
Finnish origin, wild				
IX	1.05	8.03	14.3	69 800
X	1.18	6.76	19.3	51 800
XI	0.96	7.43	15.6	64 000

values were calculated by dividing the volatile oil percentages by the threshold values (18) (Table 3).

The differences in the threshold values of the oils were relatively slight varying between 0.32 and 1.25. Although the caraway seeds collected from the wild had the highest oil contents, the corresponding threshold values were not the lowest, but only average, whether calculated on the oil or the seed itself. According to the thresholds, the aroma was strongest in samples IV and VII.

Flavour profiles of the caraway oil samples

For this assessment, the basic solutions of the oils were diluted with water to a hundredfold the threshold concentration. The strength was scored on a scale of 0–3 (definitions: 0 = no odour/taste; 1 = weak, 2 = clear, 3 = strong odour/taste). The panelists were asked themselves to define the types of odour and taste present. Number of participants was 10–12 and they were all trained.

The results are given in the following table, where the numbers in parentheses indicate the number of panelists mentioning an aroma of aniseed.

The flavour profile shows that in all the samples a typical caraway flavour was dominant, only a few of the panelists indicating a flavour of aniseed type. An interesting observation is that the typical caraway flavour was strongest in samples V and VI, which are aged. In the wild caraway the typical caraway flavour was stronger in the samples collected later (X and XI). The aftertaste was mostly bitter or oily.

Discussion

The first aim of this study was to find the most suitable methods for assessing the quality of caraway seed as a basis for quality requirements. In addition, the possibilities for developing the cultivation of caraway seed in Finland were of interest.

Characterization and assessment of the caraway oil samples by the flavour profile method

Sample	Caraway Odour	Caraway Taste	Aniseed Odour	Aniseed Taste	Other odours/tastes	Aftertaste
Foreign origin						
I	1.7	1.1	1.0 (1)	–	mint, fennel apple odour	
II	1.3	1.3	1.5 (1)	1.5 (2)	sweet, applelike, herbaceous odour, taste of rye	bitter
III	2.1	1.9	1.0 (2)	1.3 (3)	peppermint odour herbaceous taste	bitter oily
IV	1.9	1.8	1.0 (2)	1.0 (3)	sweet odour	
Finnish origin cultivated						
V	2.8	2.7	1.5 (1)	–	sweet, volatile oil odour	caraway oily
VI	2.8	2.3	1.7 (2)	1.5 (2)	sweet odour	bitter
VII	2.1	2.1	–	1.0 (1)	sweet odour taste of rye	bitter
VIII	1.5	1.4	1.5 (1)	1.0 (1)	sweet, volatile oil odour taste of rye and grass	
Finnish origin, wild						
IX	2.4	2.1	1.5 (1)	1.4 (1)	sweet odour	
X	2.7	2.4	2.0 (1)	2.0 (1)	volatile oil and fennel odour	oily
XI	2.6	2.2	1.5 (1)	1.0 (1)	volatile oil odour taste of rye	bitter

Of the methods studied, a preliminary inspection of appearance, particularly impurities, and flavour was found to give good results when trained panelists are available. The scoring system still needs to be improved. This method gives a general impression of the purity of the sample, but if necessary, the amount of foreign material can be measured by weighing. When investigating seed for cultivation, purity is studied on duplicate samples of 4 g (19). The sample size in a standardized method should be such that duplicate samples give consistent and reliable results. A preliminary inspection also gives a general impression of the strength and possible loss of the flavour.

Assays of weight loss, ash and acid-insoluble ash are customary and suitable methods by which very defective samples may be detected. In the present samples no defects were found by these methods. A useful chemical method for measuring the strength of the flavour is to assay the volatile oil content. In addition, the carvone content, obtained by gas chromatography, has an important effect on the flavour. In the samples studied the volatile oil content was highest in the wild caraway seeds, there being little variation in the carvone content.

The sensory method of threshold assessment has given information on the strength of the aroma, and flavour profile study reveals the differences between the different samples. These methods are important, since the practical value of any spice depends on the strength and purity of the flavour observed by sensory means. In the present samples the strength of the flavour showed some variation,

but these results did not correspond exactly to the strength obtained from the volatile oil content: for instance, the wild caraway had the highest volatile oil value, but the aroma was only of average strength. The flavour profile analysis, again, showed that in all the samples this characteristic was very uniform and of purely caraway type. An interesting finding was that the most typical caraway flavour was found in the aged samples.

Regarding the cultivation of caraway, it seems that in a cool climate the seeds are not always able to mature sufficiently, and in consequence the carvone content remains somewhat low (15). In the wild caraway the seeds were found to be smaller than in the cultivated samples, and the volatile oil content was clearly higher. However, the carvone content in the wild caraway seeds was not particularly high, nor was the odour threshold the lowest. In the growing of caraway, seed quality is not the only objective; several other factors are important such as earliness of maturation. yield, strength of stems, etc. (1). Although the native wild caraway certainly is well adjusted to the climate, it appears that the seed yield is not high and stems may be weak (1). It remains an open question whether cultivation would change the size and aroma content of the seeds. In conclusion, it may be worth looking into the possibilities of growing caraway of native origin, but many points still remain to be clarified.

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KÜLÖNBÖZŐ EREDETŰ KÖMÉNYMAGMINTÁK MINŐSÉGI TULAJDONSÁGAI

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A köménymag minőségi értékelésére alkalmas módszereket tanulmányozták különböző forrásokból származó 11 minta: külföldi és finn kereskedelmi minták és vadon termett finn minták alapján. A vizsgálati módszerek felölelték a szennyezésekre vonatkozó előzetes szemlét a külső megjelenés alapján a zamat és a nagyság, a súlyveszteség (nedvességtartalom), a hamu és a savban oldhatatlan hamu, az illóolaj-tartalom meghatározását és az illóolajban a karvon és limonén tartalom meghatározását gázkromatográfiával. Ezenkívül érzékszervi módszerekkel értékelték az illatok küszöbértékeit és a zamatanyagok jellegét. Tárgyalják ezeknek a módszereknek a használhatóságát. A vadon termett köménymagmintákban az illóolaj-tartalom kifejezetten nagyobb volt, mint az egyéb mintákban. A vadon termett magminták illatküszöbei azonban csak átlagosak voltak. A régi mintákban a zamatveszteség nem volt határozott, és a zamatanyagok jellege élesebben kömény típusú volt, mint a frissebb mintákban.

Tárgyalják továbbá a vad kömény termesztési lehetőségeit Finnországban.

ПОКАЗАТЕЛИ КАЧЕСТВА ТМИНА ПРОИСХОДЯЩЕГО ИЗ РАЗНЫХ ИСТОЧНИКОВ

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На основании загранияных и Финляндских торговых образцов и дикорастущих финских образцов тмина, авторы изучали метод оценки качества 11 образцов происходящих из разных источников. Методы оценки охватывали предварительный осмотр загрязненности на основании внешнего вида, определения аромата и размер, потерб веса (влагосодержание), золу и в кислоте нерастворимую золу и содержание эфирных масел, а также газохроматографическое определение содержания карвоновой и лимоневой кислоты эфирного масла. Кроме того органолептическими методами оценивали пороговые величины запаха и характер вкусовых веществ и применимость этих методов. Определили, что в дикорастущих образцах тмина содержание эфирного масла боло высше чем в прочих образцах. Пороговые значения запаха дикорастущего тмина достигали только среднюю величину. В старых образцах потеря запаха незначительна, вкусовые вещества более типичны тмину чем в свежих образцах тмина.

QUALITATIVE EIGENSCHAFTEN VON KÜMMELKÖRNERN UNTERSCHIEDLICHER HERKUNFT

T. Kuusi, J. Tenhunen, T. Hirvi und M. Suihko

Auf Grund 11 Muster unterschiedlicher Herkunft: und zwar auf Grund ausländischer und finnischer kommerziellen Muster und wild gewachsenen finnischen Muster wurden Methoden studiert, die zur Qualitätsbewertung von Kümmelkörnern geeignet sind. Die Untersuchungsmethoden umfassten eine vorangehende Besichtigung bezüglich der Verunreinigungen auf Grund der äusseren Erscheinung, ferner die Bestimmung des Aromas und der Grösse, des Gewichtsverlustes (Feuchtigkeitsgehaltes), des Aschegehaltes und der Menge der in Säure unlöslichen Asche,

die Bestimmung des ätherischen Ölgehaltes und des Gehaltes and Carvon und Limonen in ätherischen Öl durch Gaschromatographie. Ausserdem wurden die Schwellenwerte des Geruchs und der Charakter der Aromatoffe mittels sensorischen Methoden bewertet. Die Verwendbarkeit dieser Methoden wird besprochen.

In den wild gewachsenen Kümmelkörnermustern war der Gehalt an ätherischem Öl ausgesprochen höher als in den übrigen Mustern. Die Schwellenwerte des Geruchs waren jedoch in den Mustern der wild gewachsenen Kümmelkörner nur durchschnittlich. In den älteren Mustern war der Aromaverlust nicht ausdrücklich, und der Charakter der Aromastoffe wies einen schärferen Kümmeltyp auf, als in den frischeren Mustern.

Ferner werden die Anbaumöglichkeiten des wilden Kümmels in Finnland erörtert.