Short Communication

Physical Parameters, Oleoresin and Volatile Oil Contents of Five Pepper (*Pepper nigrum* L.) Cultivars as Influenced by Maturity

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Abstract: The experiment was undertaken to identify the appropriate stage of harvesting of berries to prepare quality pepper for whole use or for extraction purpose to get better oleoresin and essential oil yield. Harvesting of berries from five pepper cultivars was carried out at 3.5, 4.5, 5.5, and 6.5 months after 70% set of the berries. The experiment was arranged in a randomized complete block design with three replications at Teppi Agricultural Research Sub-center, South-western Ethiopia. Generally, peppercorn boldness and color improved with delayed harvest; 5.5 months after fruit set being the optimum. Five of the cultivars showed comparable oleoresin and volatile oil contents. The oleoresin content ranged from 13.63 to 16.01% whereas the volatile oil content ranged from 3.18 to 3.53%. Both were found to be within the acceptable ranges. The highest oleoresin (19.41%) and volatile oil (4.95%) yields were obtained from earlier harvest (3.5 months after fruit set) and decreased as harvest stages delayed. In general, it is recommended that the spikes be harvested 3.5 months after 70% fruit set to get higher oleoresin or volatile oil yield and 5.5 months after fruit set if it is intended for use as whole or in ground form.

Keywords: Black Pepper; Essential Oil; Oleoresin; Peppercorn; Volatile Oil

1. Introduction

Pepper (*Pepper nigrum* L.) in the botanical family Piperaceae is among the oldest and the most widely used spices throughout the world. Nybe and Peter (2003) pronounced that black pepper enjoys a distinction of 'King of Spices' for its varied uses and dominance in the global spice trade and is also known as "black gold" , valued for its multiple uses. It alone accounts for about 35% of the world's total spice trade (Majeed and Prakash, 2000). Major products of pepper are whole dried immature fruit (black pepper) and washed dried ripe fruit (white pepper) (Purseglove *et al.*, 1981; I-San Lin, 1994).

Black pepper is the spiciest of all spices and is the most important and authoritative of all spices. It has extensive culinary uses, and is used in meats, soups, fish, pickles and sauces, while white pepper is used primarily in cases where dark particles are undesirable, such as in light-colored sauces, mayonnaise and cream soups. The essential oils and oleoresins obtained from black pepper are mainly used for flavoring purposes; in perfumery and in pharmaceutical products (Purseglove *et al.*, 1981; Borget, 1993; I-San Lin, 1994). The quality of pepper products (black, white or green pepper) is influenced by intrinsic characteristics of the variety, the stage of maturity of berries at harvest, processing methods and conditions, and duration of storage (Pruthi *et al.*, 1976). The authors stressed that cultivar and the maturity stage of berries at harvest have a great impact on the quality of pepper, and the former can be improved by continuous selection and propagation of suitable strains. Risch (1997) also pointed out that a number of factors, including climatic conditions, can influence the abundance of the active components in spices.

The main components of product quality are type, size, shape, color, texture, composition, maturity, freedom from damage, disease, pest, and/or disorders (Jackson *et al.*, 1985). Black pepper quality is evaluated on the basis of its appearance, pungency level, aroma and flavor and the relative importance of these quality characteristics is dependent upon the intended end-use of the spice. When it is intended for direct use as a spice in whole or ground form, the appearance is of primary importance to the buyers. In general, bold-sized dried peppercorn with a uniform dark-brown to black color fetches the best prices. In contrast, the appearance of the spice has lesser importance when it is intended for processing into black pepper oleoresin or essential oil. According to Purseglove *et al.* (1981), the quality of white pepper is evaluated based on its appearance (color and size) and flavor properties (pungency and aroma).

Black pepper is used to produce oleoresin and essential oils (volatile oils) which are utilized in many ways. Oleoresin is the total pungency and flavor constituent of pepper marketed as spice drops which are in great demand in all countries (Nybe and Peter, 2003). It is produced by solvent extraction of pepper powder using a suitable organic solvent such as acetone, ethanol, ethyl acetate or ethylene dichloride, whereas black pepper oil is extracted by using steam distillation (Purseglove *et al.*, 1981; Nybe and Peter, 2003).

Diverse agro-ecology in Ethiopia allows the production of pepper particularly in the hot humid lowlands. Dry black pepper yield ranging from 1970 to 2850 kg ha^{-1} was recorded for different cultivars at Teppi Agricultural Research Sub-center. Although the crop is becoming important in the Ethiopian spice industry, the appropriate harvesting stage (time) of berries to produce quality pepper has not been identified. Hence, the current experiment was initiated with the objective of identifying the optimum harvesting stage of berries to produce quality black pepper that can be used as whole or in ground form or for extraction purposes to acquire high oleoresin and volatile oil yields.

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2. Materials and Methods

2.1. Description of the Study Site

The experiment was conducted at Teppi Agricultural Research Sub-center, located at 7° 3' N and 35° E at an altitude of 1200 m above sea level in the hot humid lowland area of south western Ethiopia. The site receives mean annual rainfall of 1750 mm and the mean minimum and maximum temperatures are 15.6 and 29.9 ^{0}C, respectively. The soil of the experimental area is generally fertile forest soil, very suitable for coffee and perennial spices production. It is grouped as Dystric Nitosol which is fertile, clay loam in texture with clay content of 30%, silt 42%, sand 28% and a pH (KCl) value of 6.8. It has an organic carbon content of 5.99%, total N of 0.98% and available P of 25.99 ppm. Furthermore, the exchangeable K content was 1.4, Ca 35.4, Mg 6.4 and cation exchange capacity was 46.8 all expressed in meq/100 gram of soil. The carbon to nitrogen ratio of the soil was 10 and percentage base saturation was 93%.

2.2. Experimental Material and Treatments

The experiment was conducted on the already established plantation of five pepper cultivars (Bra. 32/79, SR. 3/80, Pan. 4/80, Kuch. 5/80 and T_4. 228 17/79) which were arranged in randomized complete block design and replicated three times. Harvesting was done at 3.5, 4.5, 5.5, and 6.5 months after 70% fruit setting occurred. At each harvesting stage, spikes with uniform size (similar maturity stage) were picked. Berries were detached from spikes and sun-dried on stretched clothes by continuously turning over by hand until the recommended or safe moisture level (11%) was attained. At night, the samples were kept in a room to protect them from night moisture or rain.

2.3. Physical and Chemical Evaluation

After drying, 1 kg of dried berries was taken for each treatment for physical quality study, and oleoresin and volatile oil extraction. Physical parameters such as peppercorn boldness or shriveledness and color were assessed by visual observation. Samples were sent to KASSK Spices and Herbs Extraction Factory (Addis Ababa) for chemical analysis. Oleoresin yield was determined by using cleavage type apparatus and volatile oil yield was quantified by using acetone in a soxhlet apparatus.

2.4. Statistical Analysis

Data were subjected to analysis of variance (ANOVA) by the General Linear Model procedure using SAS (Statistical Analysis System, 2001). Mean comparison was performed using the least significant difference at 1% level of significance.

3. Results and Discussion

3.1. Physical Parameters

Physical observation of the samples after final drying indicated that there was great variability among samples harvested at different stages of maturity with respect to peppercorn boldness (shriveledness) and color (Table 1). Regardless of the cultivar, dried peppercorns from the first harvest (3.5 months after 70% fruit set) were too shriveled, while those from the final harvest were bold. Fruit collected 3.5 and 4.5 months after 70% fruit set developed a dull black color and delaying harvesting up to 5.5 and 6.5 months resulted in dark and dark brown colored peppercorn, respectively. From the physical observation, it was noted that harvesting 5.5 months after 70% fruit set resulted in less shriveled black colored peppercorns which are much preferred by the consumers (either ground or whole). Delaying harvesting up to 6.5 months encouraged the development of bold and dark brown peppercorn because they are extracted from spikes consisting of a high amount of red ripe berries. In most cases, however, consumers prefer black peppercorn to dark brown ones. Hence, the berries should be harvested 5.5 months after fruit set to produce less shriveled black pepper. To produce quality black pepper, the spikes should be picked as soon as one of the berries on it begins to turn red (Carlos and Balakrishnan, 1991). They also noted that picking unripe berries while green is not advisable for preparing black pepper. Purseglove *et al.* (1981) also reported that, if the spikes of pepper are left un-harvested longer (more than 6 months), the physical parameters of the peppercorn increase while extraction quality tends to decrease.

Table 1. Peppercorn boldness and color of pepper as influenced by harvest stages.

Harvesting stage (month)	Peppercorn boldness	Peppercorn color
3.5	Too shriveled	Dull black
4.5	Shriveled	Dull black
5.5	Less shriveled	Black
6.5	Bold	Dark brown

3.2. Chemical Parameters

There were no significant differences among cultivars with respect to oleoresin and volatile oil yields (Table 2). Oleoresin yield ranged from 13.63% (T_4. 228.17/79) to 16.01% (SR.3/80) while volatile oil yield varied from 3.18% (Kuch. 5/80) to 3.53% (Bra. 32/79). Richard *et al.* (1971) reported that the chemical composition of black pepper varieties varies widely. There was variation in cultivars in quality factors tested at different years-probably due to the variation in agro-climatic conditions (Menon *et al.*, 2002). From the current investigation, it has been observed that the tested cultivars can fulfill the required standard volatile oil if harvested at the right stages. According to Nurdjannah (2003), the steam volatile oil content of some cultivars of the spices could be as high as 3.8%, but for commercial scale, a reasonable quality crushed black pepper can provide yield ranging from 1 to 2.6% through steam distillation. Similarly, the oleoresin yield of the cultivars was in the recommended market standard range. Oleoresin yields of 10-13% have been reported for Indian Malabar pepper; the highest being obtained with Malabar light pepper (Nambudri *et al.*, 1970).

Table 2. Oleoresin and volatile oil yields of pepper as influenced by cultivars.

Cultivar	Oleoresin yield (%)	Volatile oil yield (%)
Bra. 32/79	15.65a	3.53a
SR. 3/80	16.01a	3.52a
Pan. 4/80	13.76a	3.35a
Kuch. 5/80	13.75a	3.18a
T_4. 228.17/79	13.63a	3.50a
SEM	0.730	0.140

Means within columns sharing the same letters are not significantly different (P < 0.01); SEM = Standard error of the mean.

Both oleoresin and volatile oil yields were significantly influenced by harvesting stage (Table 3). Regardless of the cultivars, the highest yields of oleoresin (19.41%) and volatile oil (4.95%) were obtained from peppercorns harvested 3.5 months after fruit set. Harvesting stage was negatively correlated with oleoresin (r=-0.98**) and volatile oil (r=-0.99**) yield indicating that there was a progressive decline both in oleoresin and volatile oil yields in response to delaying harvesting. The volatile oil of immature green pepper reaches a maximum at early stage (4.5 months after fruit setting) for some varieties in India, and diminishes while the piperine content continues to increase for some period (GCSSI, 2003). Similarly, Purseglove *et al.* (1981) reported that pepper quality is significantly influenced by harvesting stage and the highest and lowest volatile oil yields of 10.4 and 3.6% (v/w) were recorded from peppercorn harvested 4.5 and 7.0 months after fruit set, respectively. Jansz *et al.* (2006) also reported most oil synthesis in black pepper appeared to have taken place by 23 weeks. It has been generalized that, for most of the spices, earlier harvesting is recommended to attain a good volume of extraction yield with the exception that high volume of volatile oil yield per hectare bases will be obtained from late harvesting of ginger (Purseglove *et al.*, 1981; KAU, 2002).

Table 3. Oleoresin and volatile oil yields of pepper as influenced by harvest stage.

Harvesting stage (month)	Oleoresin yield (%)	Volatile oil yield (%)
3.5	19.41a	4.95a
4.5	16.72b	3.66b
5.5	11.74c	2.89c
6.5	10.37c	2.15d
SEM (±)	0.650	0.130

Means within columns sharing the same letters are not significantly different (P < 0.01); SEM = Standard error of the mean.

4. Conclusions

The five cultivars exhibited comparable oleoresin as well as volatile oil contents. However, the harvesting stage of the spikes influenced to a grate extent the quality parameters of pepper, such as peppercorn, boldness and color and oleoresin and volatile oil yield. Early harvest (3.5 months after fruit set) gave the highest oleoresin and volatile oil yields while harvesting 5.5 months after fruit set resulted in less shriveled black peppercorns which are much preferred for whole or crushed home-use. Hence, the purpose of pepper production must be clearly known and the harvesting period should be adjusted accordingly.

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