



## Nutrient accumulation in fruits and grains of black pepper at different ripening stages

Jéssica Rodrigues Dalazen<sup>1</sup>  Gustavo Pereira Valani<sup>2</sup>  Henrique Duarte Vieira<sup>3</sup>   
José Cochicho Ramalho<sup>4,5</sup>  Valdemar Lacerda Jr.<sup>6</sup>  Wanderson Romão<sup>7</sup>  Fábio Luiz Partelli<sup>8\*</sup> 

<sup>1</sup>Rede BIONORTE, Universidade Federal de Rondônia (UNIR), Porto Velho, RO, Brasil.

<sup>2</sup>Universidade de São Paulo (USP), Piracicaba, SP, Brasil.

<sup>3</sup>Universidade Estadual do Norte Fluminense Darcy Ribeiro (UENF), Campos dos Goytacazes, RJ, Brasil.

<sup>4</sup>PlantStress & Biodiversity Lab, Centro de Estudos Florestais (CEF), Instituto Superior Agronomia (ISA), Universidade de Lisboa (ULisboa), Oeiras, Portugal.

<sup>5</sup>Unidade de Geobiociências, Geoengenharias e Geotecnologias (GeoBioTec), Faculdade de Ciências e Tecnologia (FCT), Universidade NOVA de Lisboa (UNL), Caparica, Portugal.

<sup>6</sup>Universidade Federal do Espírito Santo (UFES), Vitória, ES, Brasil.

<sup>7</sup>Instituto Federal do Espírito Santo (IFES), Vila Velha, ES, Brasil.

<sup>8</sup>Universidade Federal do Espírito Santo (UFES), 29932-540, São Mateus, ES, Brasil. E-mail: [partelli@yahoo.com.br](mailto:partelli@yahoo.com.br).

**ABSTRACT:** *Nutrients accumulation in plants/fruits varies according to the crop development stage and its quantification is important to determine nutrients exportation for fertilization planning and nutrient balancing. This study determined the accumulation of nutrients in different parts of Piper nigrum 'Bragantina' spikes harvested at two ripening stages (still-green and red-colored spikes). Assessments in separate parts of still-green spikes (fruits and peduncles) as well as in red-colored spikes (grains, flesh and peduncles) were performed. Piper nigrum spikes were harvested, dried, and the parts were thereafter separated for chemical analysis. N and Mn were the macro and micronutrient most accumulated in the fruits of still-green spikes and in the grains of red-colored spikes, while K and B were most accumulated in the other parts of black pepper assessed, regardless of the ripening stage. The process of removing the flesh from the fruits for white pepper production led to a difference in nutrient concentration and accumulation between the fruits of still-green spikes and the grains of red-colored spikes. A significant contribution of nutrient input can be achieved by maintained flesh and peduncles in the crop area, decreasing the dependence of external fertilizers and thus contributing to a more sustainable agriculture.*

**Key words:** *Piper nigrum, macronutrients, micronutrients, white pepper.*

## Acúmulo de nutrientes em frutos e grãos de pimenta-do-reino em diferentes estádios de maturação

**RESUMO:** *O acúmulo de nutrientes em plantas/frutos variam de acordo com o estágio de desenvolvimento das culturas, sendo que sua quantificação é importante na determinação da exportação de nutrientes para o planejamento da adubação e para o balanceamento de nutrientes. Esse estudo objetivou determinar o acúmulo de nutrientes em diferentes partes dos cachos de Piper nigrum 'Bragantina' colhidos em dois estágios de maturação (coloração da casca verde e coloração da casca vermelha). As avaliações foram feitas separadamente nos cachos de Piper nigrum com coloração verde (fruto e pedúnculo) e vermelha (grão, casca e pedúnculo), em que os cachos foram colhidos, secos e as partes foram separadas para análises químicas. N e Mn foram os macro e micronutrientes mais acumulados nos frutos dos cachos com coloração da casca verde e nos grãos dos cachos com coloração da casca vermelha, enquanto K e B foram mais acumulados em outras partes da planta, independente do estágio de maturação. O processo de remoção da casca para produção de pimenta branca levou a uma diferença na concentração e no acúmulo de nutrientes nos frutos de pimenta preta e nos grãos de pimenta branca. Uma contribuição significativa para entrada de nutrientes pode ser obtida se as cascas e pedúnculos forem mantidos na área de cultivo, o que diminui a dependência de fertilização, contribuindo para uma agricultura mais sustentável.*

**Palavras-chave:** *Piper nigrum, pimenta preta, macronutrientes, micronutrientes, pimenta branca.*

## INTRODUCTION

Black pepper (*Piper nigrum* L.) is a crop of global importance. It is used in gastronomy, as seasoning in the preparation of industrialized foods; in the pharmaceutical industry, for the composition of medicines; and in agriculture, as a natural insecticide

due to its allelopathic compounds (CHIN ANN, 2016; RAVINDRAN, 2003).

*Piper nigrum* production is economically and socially important for Brazil. In relation to the economic importance, its export trade in the country reached values of about US\$ 346 million in 2016 (INTERNATIONAL PEPPER COMMUNITY, 2018).

Considering both its economic and social impacts, it is important to note that most black pepper growing areas in the country are owned by smallholder farms, which are cultivated to supplement the farm income. Furthermore, *Piper nigrum* cultivation requires high rates of labor in the field, leading to a greater income distribution, promoting a higher quality of life in rural areas, and reducing rural exodus (PARTELLI, 2009).

*Piper nigrum* byproducts are reported in many different forms in the world market, from black and white pepper grains – which differ from one another by the harvesting stage and processing method – to products already ground and sold in small packages. In order to obtain black pepper, for example, the spikes are harvested when the spikes are still-green. The browning of unripe still-green peppercorns is attributed to several polyphenolic compounds that become enzymatically oxidized (RAVINDRAN, 2003). White pepper, conversely, is obtained by harvesting the spikes when peppercorns are already red-colored, followed by the removal of the fruits flesh (pericarp/wall), and only the grains are dried, making this a less aromatic and a more delicate pepper (BAKER & GRANT, 2019).

In relation to nutrient accumulation, PADUIT et al. (2018) reported that between different parts of black pepper, the fruits concentrated most macronutrients in other plant parts, such as roots, stems, braches and leaves and fruits. It is important to note that the accumulation of important constituents in *Piper nigrum* spikes, such as starch, volatile oils, ethereal non-volatile extract and piperine, is known to be influenced by its ripening stage, as well by the crop cultivar, and it is not regularly distributed in different parts of the spikes (HUSSAIN et al., 2017). Furthermore, as black pepper is cultivated in different agroecological situations, site-specific studies are needed, as suggested by a review about the agronomy of black pepper (SIVARAMAN et al., 1999), which is important to address different conditions, such as different soil groups and fertility status (SRINIVASAN et al., 2007).

Considering that i) *Piper nigrum* demand for nutrients varies according to its development stage, ii) the reproductive phase is the period of greatest nutrient extraction, and iii) most nutrients absorbed by the plant are concentrated in the fruits; a precise information about the quantities of nutrients in different parts of *Piper nigrum* spikes harvested from different ripening stages is crucial for determining the removal of nutrients from the crop area accordingly. Thus, this study determined the accumulation of nutrients in different parts of still-green spikes (fruits

and peduncles) and red-colored spikes (grains, flesh and peduncles) of *Piper nigrum* L.

## MATERIALS AND METHODS

### Study Site

The study site was located in the municipality of São Mateus, northern Espírito Santo State (18°46'48.4"S, 39°52'31.5"W and 23 m above sea level). According to Köppen's classification, the climate of the region is Aw, tropical with dry season during winter and rainy summer (ALVARES et al., 2013).

Spikes of *Piper nigrum* 'Bragantina' were harvested in March 2018 (three-year-old field). The crop was grown under full sun, with 3.5 x 1.8 m spacing in a Ferralsol (IUSS WORKING GROUP WRB, 2015). Physical (GEE & BAUDER, 2002) and chemical (TEIXEIRA et al., 2017) characteristics of the soil in the 0 - 20 cm layer are as follow:  $\text{pH}_{\text{water}}$  (1:2.5 v v<sup>-1</sup>) = 5.05; P (Mehlich-1) = 52 mg dm<sup>-3</sup>; K = 95 mg dm<sup>-3</sup>; S = 8 mg dm<sup>-3</sup>; Ca<sup>2+</sup> = 4 cmolc dm<sup>-3</sup>; Mg<sup>2+</sup> = 0.35 cmolc dm<sup>-3</sup>; Al<sup>3+</sup> = 0.5 cmolc dm<sup>-3</sup>; titratable acidity (H+Al) = 5.9 cmolc dm<sup>-3</sup>; organic matter = 2.2 dag dm<sup>-3</sup>; sum of bases = 4.62 cmolc dm<sup>-3</sup>; cation exchange capacity = 5.12 cmolc dm<sup>-3</sup>; sand content = 679.5 g kg<sup>-1</sup>; silt content = 60.5 g kg<sup>-1</sup>; and clay content = 260 g kg<sup>-1</sup>.

Within farming management practices, weeds were controlled with herbicide. Lime and fertilizer were applied based on a soil analysis and regional recommendations (PREZOTTI et al., 2007). The field was irrigated by drip irrigation and pruning were performed periodically. The crop received 400 kg ha<sup>-1</sup> year<sup>-1</sup> of N, 80 kg ha<sup>-1</sup> year<sup>-1</sup> of P<sub>2</sub>O<sub>5</sub> and 320 kg ha<sup>-1</sup> year<sup>-1</sup> of K<sub>2</sub>O, distributed over each year.

### Spikes sampling and nutrient analysis

Two ripening stages of *Piper nigrum* spikes were assessed. The first was composed of still-green spikes, whereas two separate parts of the spikes (fruits and peduncles) were assessed (Figure 1). The second condition was composed of red-colored spikes and three separate parts of the spikes (grains, flesh and peduncles) were assessed (Figure 2). Each ripening stage was considered as a different experiment in this study, wherein the experimental design was completely randomized, with nine spikes and three replicated per experiment.

Each part of the spike was weighted for fresh and dry mass. Both were measured on a precision scale (0.001 g). Dry mass was weighted after the samples were dried in a forced ventilation oven at 70 °C for 72 hours. Additionally, samples were dried at 105 °C for

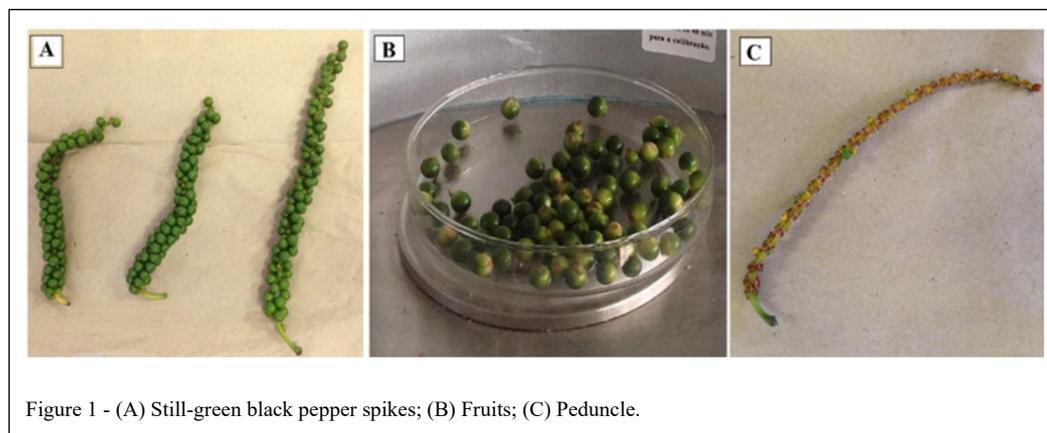


Figure 1 - (A) Still-green black pepper spikes; (B) Fruits; (C) Peduncle.

24 hours and the final dry mass and nutrient export were adjusted to the moisture of 12 %, which is the moisture value at which black pepper is sold.

The concentration of N, P, K, Ca, Mg, S, Fe, Zn, Cu, Mn and B in the spikes were assessed according to the methodology described by MALAVOLTA et al. (1997), which can be summarized as follows: N was assessed by the semi-micro Kjeldahl method, P by colorimetric determination with metavanadate, K by flame photometry, Ca and Mg by EDTA complexometric titration, S by barium sulphate gravimetry, and micronutrients by inductively coupled plasma optical emission spectrometry. The accumulation of nutrients in different parts of the spike was calculated based on the dry mass x the concentration of each nutrient.

#### *Data analysis*

The resulting data for dry mass, nutrient concentration and accumulation in different parts for still-green spikes (fruits and peduncles) and for red-colored spikes (grains, flesh and peduncles) were submitted to analysis of variance by the F test ( $P < 0.01$ ). The means were compared with the Tukey's test ( $P < 0.05$ ), using the statistical program Sisvar 5.6 (FERREIRA, 2011).

## RESULTS AND DISCUSSION

#### *Dry mass and nutrient concentration*

Dry mass differed among parts of the spike for both ripening stages (Table 1). The parts that are actually sold, fruits (from black pepper) and grains (from white pepper), were responsible for approximately 95% and 68% of the dry mass production, respectively. Thus, vegetal waste is

generated from both conditions, in which black pepper production generates approximately 5% waste and white pepper 32%, whereas most comes from the flesh (Table 1). Such waste is useful for making sustainable bio-composite that could be used to replace traditional plastic (HOLILAH et al., 2021).

Most nutrients concentration differed ( $P < 0.05$ ) between spike parts for both ripening stages (Table 2). The difference in macronutrient concentration in the fruits and peduncles of still-green spikes were significant for all nutrients, except for Mg and S (Table 2). In such condition (still-green spikes) the highest concentration of N and P were found in the fruits and K and Ca in the peduncle. Considering the red-colored spikes, higher concentration of K and Mg and lower N concentration were found in both flesh and peduncles in relation to the grains. In such condition (red-colored spikes), Ca concentration was higher in the peduncles, which showed the lowest P concentration, although it did not differ from the P concentration in the flesh. Furthermore, the S concentration did not differ significantly with different parts of the spikes (Table 2). In both conditions, the macronutrients concentrations followed the order:  $N > K > Ca > Mg > P > S$ , which are in agreement with order of macronutrient requirement for black pepper (VELOSO & CARVALHO, 1999).

Regarding micronutrient concentration in the still-green spikes, it is observed that Mn concentration did not differ between fruits and peduncles, differently from all other micronutrients. In such condition (still-green spikes), higher Cu concentration was reported in the fruits; and higher Fe, Zn and B concentration in the peduncles (Table 2). Considering the red-colored spikes, Cu concentration did not differ between the assessed parts. Moreover, peduncles concentrated most Fe and Zn. The Mn

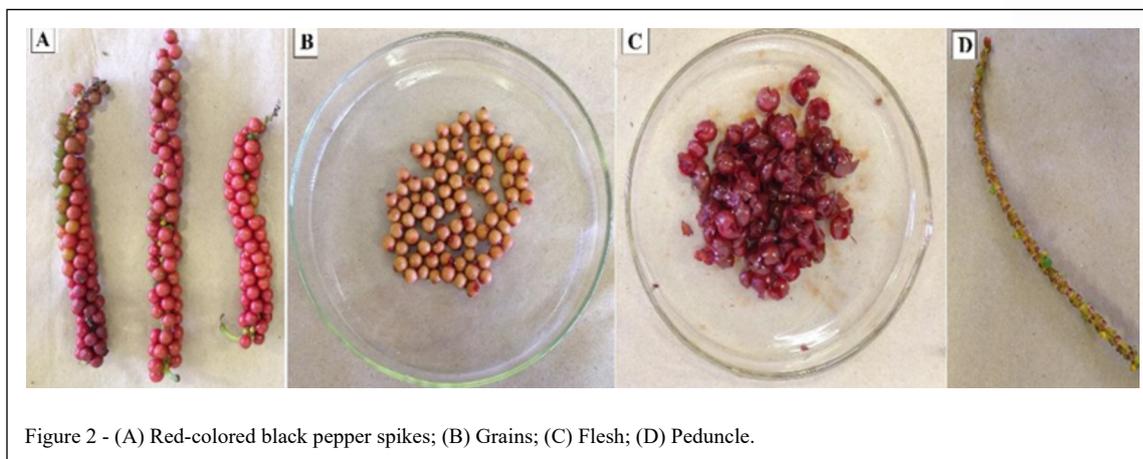


Figure 2 - (A) Red-colored black pepper spikes; (B) Grains; (C) Flesh; (D) Peduncle.

concentration was not different between peduncles and grains, being higher than the concentration in the flesh. B concentration, on its turn, was not different between flesh and peduncles, being higher than its concentration in the grains (Table 2). Although assessments of micronutrients in reproductive parts of black pepper are scarce, the results of this study greatly vary from other works (BHAT et al., 2010; PRADEEP et al. 1993), which may be a result of biotic and abiotic factors, such as climate, crop cultivar and soil composition (SALEH-E-IN et al., 2017).

A study assessing the chemical quality of fruits of two cultivars of *Piper nigrum* – one being the Indigenous cultivar, sampled from the Bangladesh Tea Research Institute; and the other, Kerala,

sampled from the market of Indian spices in Kerala – found significantly higher Fe (138.36 and 344.07 mg kg<sup>-1</sup>, respectively) and K (13.96 and 20.77 g kg<sup>-1</sup>, respectively) concentration than those obtained with the Bragantina cultivar in this study. Furthermore, Ca and Mg concentration were similar to those presented here (ABUKAWSAR et al., 2018).

High quantities of nutrients are lost during the process of separating flesh from the fruits in white pepper production, especially K, Mg, B, Fe and Zn. This leads to the chemical differences between black and white pepper, which may also alter the sensorial characteristics of the final product (JAGELLA & GROSCH, 1999). The flesh and peduncles of *Piper nigrum* are by-products generated during pepper

Table 1 - Fresh and dry mass of separate parts for still-green spikes (fruits and peduncles) and red-colored spikes (grains, flesh and peduncles).

-----Still-green spikes-----		
Part	Fresh mass (g)	Dry mass (g)
Fruits	14.77 a	4.97 a
Peduncles	1.68 b	0.27 b
CV %	11.92	4.24
-----Red-colored spikes-----		
Part	Fresh mass (g)	Dry mass (g)
Grains	6.061 a	3.78 a
Flesh	6.95 a	1.45 b
Peduncles	1.55 b	0.30 c
CV %	13.17	21.09

CV: coefficient of variation. Means followed by the same letter in the column do not differentiate between each other according to Tukey's test ( $P < 0.05$ ).

Table 2 - Nutrient concentration in separate parts of still-green spikes (fruits and peduncles) and red-colored spikes (grains, flesh and peduncles).

-----Still-green spikes-----						
Parts	-----Macronutrients (g kg <sup>-1</sup> )-----					
	N	P	K	Ca	Mg	S
Fruits	20.11 a	1.52 a	11.04 b	9.65 b	1.65 a	0.99 a
Peduncles	16.12 b	0.87 b	22.29 a	14.54 a	1.62 a	0.84 a
CV (%)	5.14	8.16	5.72	9.92	4.91	14.88
Parts	-----Micronutrients (mg kg <sup>-1</sup> )-----					
	Fe	Zn	Cu	Mn	B	
Fruits	36.67 b	18.67 b	9.33 a	38.00 a	25.33 b	
Peduncles	71.33 a	59.00 a	6.00 b	39.00 a	72.33 a	
CV (%)	17.36	17.78	10.65	6.87	17.77	
-----Red-colored spikes-----						
Parts	-----Macronutrients (g kg <sup>-1</sup> )-----					
	N	P	K	Ca	Mg	S
Grains	21.82 a	1.37 a	4.17 b	11.19 b	0.83 b	0.99 a
Flesh	12.93 b	1.10 ab	18.34 a	9.94 b	1.94 a	0.72 a
Peduncles	14.54 b	0.87 b	19.66 a	14.68 a	1.80 a	0.84 a
CV (%)	7.77	10.79	9.99	9.77	12.28	17.53
Parts	-----Micronutrients (mg kg <sup>-1</sup> )-----					
	Fe	Zn	Cu	Mn	B	
Grains	14.67 c	13.67 b	7.33 a	38.00 a	16.00 b	
Flesh	37.00 b	14.67 b	7.67 a	14.00 b	63.33 a	
Peduncles	69.67 a	45.67 a	5.67 a	39.67 a	78.00 a	
CV (%)	20.57	21.96	18.74	17.86	14.78	

CV: coefficient of variation. Means followed by the same letter in the column do not differentiate between each other according to Tukey's test ( $P < 0.05$ ).

processing, which are often discarded on farmyards. From this perspective, considering its significant concentration of nutrients, such by-products may be used as fertilizers, similarly to coffee straw and other crops residues used.

#### Nutrient accumulation

The accumulation of nutrients in different parts of the still-green spikes were significant different ( $P < 0.05$ ) for all nutrients, wherein most of nutrients were accumulated in fruits (Table 3). This is related of higher dry mass of the fruits, which accounted for 95% of the spike dry mass (Table 1), which is common for pepper (SUPALKOVA et al., 2007). In such condition (still-green spike), the most accumulated nutrients in the fruits were N, K, Ca, Mg, P, S, Mn, Fe, B, Zn and Cu.

The accumulation of nutrients in different parts of the red-colored spikes were predominantly significant ( $P < 0.05$ ), except for Fe (Table 3). In the grains, which is the most economic important part,

the highest means of nutrient concentration were found for N, Ca, P, S, Mn, Zn and Cu. The iron accumulation, for example, is an important source for this micronutrient in relation to its bioavailability for human nutrition (PLATEL & SRINIVASAN, 2016). Moreover, Mg accumulation in grains was similar to flesh and highest K and B concentration were reported in the flesh. The peduncles, in its turn, was the portion of the spike with lowest nutrient accumulation, even when not significant.

According to the results presented in Table 3, N, K and Ca were the macronutrients with greater accumulation in the distinct spikes parts at both ripening stages. The nutrient reported in greater abundance was N. COVRE et al. (2016) found that, as in *Piper nigrum* grains, N concentration in coffee grains are higher than in the flesh. It is important to note that plants need adequate N levels, as the nutrient constitute several cellular components, such as amino acids, nucleic acids and hormones, which are crucial for fruits formation and fruits growth. Thereafter,

Table 3 - Nutrient accumulation in separate parts of still-green spikes (fruits and peduncles) and red-colored spikes (grains, flesh and peduncles).

-----Still-green spikes-----						
Parts	-----Accumulation of macronutrients (mg spike <sup>-1</sup> )-----					
	N	P	K	Ca	Mg	S
Fruits	100.04 a	7.56 a	54.92 a	48.01 a	8.18 a	4.92 a
Peduncles	4.38 b	0.24 b	6.08 b	3.94 b	0.44 b	0.24 b
CV (%)	7.82	10.72	6.04	6.71	5.94	8.30
Parts	-----Accumulation of micronutrients (µg spike <sup>-1</sup> )-----					
	Fe	Zn	Cu	Mn	B	
Fruits	182.13 a	92.45 a	46.47 a	189.20 a	126.47 a	
Peduncles	19.08 b	15.99 b	1.64 b	10.77 b	19.59 b	
CV (%)	23.84	22.85	12.11	12.74	28.49	
-----Red-colored spikes-----						
Parts	-----Accumulation of macronutrients (mg spike <sup>-1</sup> )-----					
	N	P	K	Ca	Mg	S
Grains	82.75 a	5.20 a	15.12 b	42.21 a	3.18 a	3.73 a
Flesh	18.74 b	1.60 b	26.63 a	14.34 b	2.78 a	1.04 b
Peduncles	4.41 b	0.26 b	5.20 c	4.46 b	0.55 b	0.26 b
CV (%)	27.41	30.79	21.61	22.47	24.59	26.38
Parts	-----Accumulation of micronutrients (µg spike <sup>-1</sup> )-----					
	Fe	Zn	Cu	Mn	B	
Grains	56.48 a	50.89 a	27.77 a	141.33 a	58.91 b	
Flesh	54.42 a	21.34 b	11.17 b	20.33 b	91.00 a	
Peduncles	21.13 a	13.87 b	1.72 b	12.04 b	23.70 c	
CV (%)	42.36	26.06	32.68	26.13	14.40	

CV: coefficient of variation. Means followed by the same letter in the column do not differentiate between each other according to Tukey's test ( $P < 0.05$ ).

severe N deficiency drastically restricts plant growth (TAIZ et al., 2014) and limits the development of pepper (VIÉGAS et al., 2013).

Similarly to other nutrients, K was not evenly distributed within the different spikes parts. This nutrient was reported in higher quantity in the flesh than in the grains, corroborating COVRE et al. (2016), who studied robusta coffee beans. Potassium is known as an essential element in the enzymatic regulation of respiration and photosynthesis, as well as in the synthesis of nitrogen compounds, such as proteins (TAIZ et al., 2014). Magnesium, as well as phosphorus and sulfur, were found to be less abundant in the parts of *Piper nigrum* spikes (Table 3). However, these nutrients are crucial for plant metabolism. P, for example, is stored in seeds as phytic acid, acting on embryo development, seed germination and seedling growth (PANDEY, 2015).

Regarding micronutrients, Mn was the most accumulated micronutrient in the grain, followed by B and Fe (Table 3). B was more accumulated in

the flesh. These results differed from those found by COVRE et al. (2016), in which the micronutrient most accumulated in coffee grains was Fe, followed by Cu and B; and for the flesh, the most accumulated micronutrient was Fe, followed by B. Although Fe accumulation did not differ among the parts of red-colored spikes, this nutrient was abundant in their grains, flesh and peduncles. Similar results were observed in robusta coffee by BRAGANÇA et al. (2007), who reported that Fe was the most abundant micronutrient in flesh, grains and in the fruits as a whole. Such results may be attributed to its important role in photosynthesis and biosynthesis of proteins and chlorophyll (TAIZ et al., 2014), as well as being a constituent of several different proteins such as hemoproteins and iron-sulfur proteins, and enzymes such as lipoxigenases (BRAGANÇA et al., 2007).

#### *Nutrients exported during harvesting*

In order to obtain a ton (1000 kg) of pepper fruits and grains, 1055 kg of dried still-green spikes

are required, or 1436 kg of red-colored dried spikes if the flesh is removed (Table 4). From this total, 55 to 80 kg of vegetable waste is generated as the peduncles are not used. Such loss is even higher when the fruit flesh is removed, exceeding 31% (463.13 kg) of flesh and peduncle, by-products that are generally discarded. Such waste may be used for compost, which is known to improve pepper yield and the system sustainability (YU et al., 2019).

It is important to note that *Piper nigrum* final products export a considerable amount of some nutrients, which may be returned to the soil in order to maintain or enhance soil quality and the crop productivity by promoting favorable effects on soil properties and life-support processes (LAL, 2008), including the input of nutrients to the plants. Otherwise, the soil will impoverish at each crop cycle and lose its sustainability with the export of nutrients via the harvest of pepper spikes.

Among nutrients, N was the most exported in both still-green and red-colored spikes. The export of N can be minimized by returning unsold parts of the spike to the crop area, which would be more relevant for white pepper production, since it would return 5.38 kg of N for every 1000 kg of harvested

spike, reducing the export of N in 21.9% by returning the flesh and peduncles to the soil. Such management would contribute into a more sustainable fertilizer planning, decreasing the need for mineral fertilizer (SCHILS et al., 2020).

After N, K and Ca were the following two most exported nutrients. In the flesh of red-colored spikes, 67.3% of K may return to the soil whether the harvested flesh and peduncles are maintained in the area. The return of Ca via flesh and peduncles is lower than N return, and thereafter most Ca should return to the soil by means of fertilization and/or liming (Table 4). In still-green spikes, nutrient cycling is lower as only the peduncle can be returned to the soil and most nutrients will be exported with the fruit. In this case, most of the exported nutrients should be replenished by means of fertilization and liming. Further studies should investigate the effect of returning pepper waste into the soil and its influence on the soil fertility status, especially because composts from other crops are known to improve the nutritional quality and pepper yields (ÇERÇİOĞLU, 2019).

Regarding micronutrients, Mn was the most exported nutrients by fruits and grains, followed by Fe and B. Fe and B were also the most abundant

Table 4 - Nutrients exported in 1000 kg of black pepper harvested at 12% moisture.

-----Still-green spikes-----							
Parts	kg	-----Macronutrients (kg)-----					
		N	P	K	Ca	Mg	S
Fruits	1000	17.70	1.34	9.72	8.49	1.45	0.87
Peduncles	55	0.78	0.04	1.08	0.70	0.08	0.04
Spikes	1055	18.48	1.38	10.80	9.20	1.53	0.91
Parts	kg	-----Micronutrients (g)-----					
		Fe	Zn	Cu	Mn	B	
Fruits	1000	32.27	16.43	8.21	33.44	22.29	
Peduncles	55	3.45	2.86	0.29	1.89	3.50	
Spikes	1055	35.72	19.28	8.50	35.33	25.79	
-----Red-colored spikes-----							
Parts	kg	-----Macronutrients (kg)-----					
		N	P	K	Ca	Mg	S
Grains	1000	19.20	1.21	3.67	9.85	0.73	0.87
Flesh	382.68	4.35	0.37	6.18	3.35	0.65	0.24
Peduncles	80.45	1.03	0.06	1.39	1.04	0.13	0.06
Spikes	1463.13	24.58	1.64	11.24	14.23	1.51	1.17
Parts	kg	-----Micronutrients (g)-----					
		Fe	Zn	Cu	Mn	B	
Grains	1000	12.91	12.03	6.45	33.44	14.08	
Flesh	382.68	12.46	4.94	2.58	4.71	21.33	
Peduncles	80.45	4.93	3.23	0.40	2.81	5.52	
Spikes	1643.13	30.30	20.20	9.44	40.96	40.93	

nutrients in the flesh and peduncles of both still-green and red-colored spikes. Thus, a great amount of these micronutrients may return to the soil if flesh and peduncles are returned to the field, which is important because organic fertilization are known to increase the extractable micronutrients in the soil compared with mineral fertilization (HERENCIA et al., 2008). Similarly, part of the micronutrients exported in the fruits and grains should return to the soil via fertilization (Table 4).

## CONCLUSION

This study assessed the concentration, accumulation and export of nutrients of *Piper nigrum* harvested in two different ripening stages. Nitrogen was the most accumulated nutrient in the fruits of still-green spikes and in the grains of red-colored spikes, while K was the nutrient most accumulated in the other parts of black pepper assessed, regardless of the ripening stage. Similarly, Mn was the micronutrient most accumulated in the fruits of still-green spikes and in the grains of red-colored spikes, while B was most accumulated in the other parts. The process of removing the flesh from the fruits for white pepper production led to a difference in nutrient concentration and accumulation between the fruits of still-green spikes and the grains of red-colored spikes.

A significant contribution of nutrient input can be achieved by maintained flesh and peduncles in the crop area, decreasing the dependence of external fertilizers and thus contributing to a more sustainable agriculture. The results of this study may support future recommendations and adjustments to black pepper fertilization as it contributed to promote a better understanding nutrients exports for *Piper nigrum*.

## ACKNOWLEDGEMENTS

This work received funding support from Fundação de Amparo à Pesquisa e Inovação do Espírito Santo (FAPES), from Coordenação de Aperfeiçoamento de Pessoal de Nível Superior (CAPES), Brasil - Finance code 001, from Conselho Nacional de Desenvolvimento Científico e Tecnológico (CNPq), and from Fundação para a Ciência e a Tecnologia, I.P. (FCT), Portugal, through the research units UIDB/00239/2020 (CEF), and UIDP/04035/2020 (GeoBioTec). We would like to acknowledge the Universidade Federal do Espírito Santo (UFES) for providing the facilities needed and the farmer Zenor Quinquim for field support.

## DECLARATION OF CONFLICT OF INTEREST

The authors declare no conflict of interest.

## AUTHORS' CONTRIBUTIONS

All authors contributed equally for the conception and writing of the manuscript. All authors critically revised the manuscript and approved of the final version.

## REFERENCES

- ABUKAWSAR, M. M. et al. Chemical, pharmacological and nutritional quality assessment of black pepper (*Piper nigrum* L.) seed cultivars. **Journal of Food Biochemistry**, v.42, n.6, p.e12590, 2018. Available from: <<https://doi.org/10.1111/jfbc.12590>>. Accessed: Oct. 29, 2019. doi: 10.1111/jfbc.12590.
- ALVARES, C. A. et al. Köppen's climate classification map for Brazil. **Meteorologische Zeitschrift**, v.22, n.6, p.711–728, 2013. Available from: <<https://doi.org/10.1127/0941-2948/2013/0507>>. Accessed: Sep. 14, 2020. doi: 10.1127/0941-2948/2013/0507.
- BAKER, B. P. & GRANT, J. A. **White pepper profile**: Active ingredient eligible for minimum risk pesticide use. Geneva: New York State Integrated Pest Management and Cornell Cooperative Extension, 2019. Available from: <<https://hdl.handle.net/1813/56144>>. Accessed: May, 3, 2020.
- BHAT, R. et al. Determination of mineral composition and heavy metal content of some nutraceutically valued plant products. **Food Analytical Methods**, v.3, n.3, p.181–187, 2010. Available from: <<https://doi.org/10.1007/s12161-009-9107-y>>. Accessed: Apr. 15, 2021. doi: 10.1007/s12161-009-9107-y.
- BRAGANÇA, S. M. et al. B, Cu, Fe, Mn and Zn accumulation by Conilon coffee plant. **Revista Ceres**, v.54, n.314, p.398–404, 2007. Available from: <<http://www.ceres.ufv.br/ojs/index.php/ceres/article/view/3382>>. Accessed: Oct. 29, 2019.
- ÇERÇİOĞLU, M. Compost effects on soil nutritional quality and pepper (*Capsicum annum* L.) yield. **Journal of Agricultural Sciences**, v.25, p.155–162, 2019. Available from: <<https://doi.org/10.15832/ankutbd.396547>>. Accessed: Aug. 30, 2021. doi: 10.15832/ankutbd.396547.
- CHIN ANN, Y. Efficacy of organic products as black pepper foliar fertilizer. **International Journal of Environment, Agriculture and Biotechnology**, v.1, n.3, 2016. Available from: <<https://dx.doi.org/10.22161/ijeab/1.3.42>>. Accessed: Mar. 5, 2020. doi: 10.22161/ijeab/1.3.42.
- COVRE, A. M. et al. Nutrient accumulation in bean and fruit from irrigated and non-irrigated *Coffea canephora* cv. Conilon. **Emirates Journal of Food and Agriculture**, v.28, n.6, p.402–409, 2016. Available from: <<https://doi.org/10.9755/ejfa.2016-04-341>>. Accessed: Jul. 21, 2020. doi: 10.9755/ejfa.2016-04-341.
- FERREIRA, D. F. Sisvar: a computer statistical analysis system. **Ciência e Agrotecnologia**, 2011. v.35, n.6, p.1039–1042. Available from: <<https://doi.org/10.1590/S1413-70542011000600001>>. Accessed: Apr. 21, 2019. doi: 10.1590/S1413-70542011000600001.
- GEE, G.; BAUDER, J. Particle-size analysis. In: DANE, J. & TOPP, G. **Methods of soil analysis: Part 4 physical methods**. Madison: Soil Science Society of America, 2002, p.255–293.
- HERENCIA, J. F. et al. The effect of organic and mineral fertilization on micronutrient availability in soil. **Soil Science**,

- v.173, n.1, p.69–80, 2008. Available from: <<https://doi.org/10.1097/ss.0b013e31815a6676>>. Accessed: Aug. 30, 2021. doi: 10.1097/ss.0b013e31815a6676.
- HOLILAH, H. et al. Hydrothermal assisted isolation of microcrystalline cellulose from pepper (*Piper nigrum* L.) processing waste for making sustainable bio-composite. **Journal of Cleaner Production**, v.305, p.127229, 2021. Available from: <<https://doi.org/10.1016/j.jclepro.2021.127229>>. Accessed: Aug. 30, 2021. doi: 10.1016/j.jclepro.2021.127229.
- HUSSAIN, Z. et al. Isolation and evaluation of piperine from black pepper and white pepper. **World Journal of Pharmacy and Pharmaceutical Sciences**, v.6, n.8, p.1424, 2017. Available from: <<https://doi.org/10.20959/wjpps20178-9752>>. Accessed: Mar. 5, 2020. doi: 10.20959/wjpps20178-9752.
- INTERNATIONAL PEPPER COMMUNITY. **Pepper statistical yearbook**. Jakarta: IPP, 2018. Available from: <<http://www.ipcnet.org/n/psy2018/>>. Accessed: Mar. 16, 2019.
- IUSS WORKING GROUP WRB. **World reference base for soil resources: International soil classification system for naming soils and creating legends for soil maps**. Rome: FAO, 2015. Available from: <<http://www.fao.org/soils-portal/data-hub/soil-classification/world-reference-base/en/>>. Accessed: Jun. 20, 2019.
- JAGELLA, T. & GROSCHE, W. Flavour and off-flavour compounds of black and white pepper (*Piper nigrum* L.): Evaluation of potent odorants of black pepper by dilution and concentration techniques. **European Food Research and Technology**, v.209, n.1, p.16–21, 1999. Available from: <<https://doi.org/10.1007/s002170050449>>. Accessed: Aug. 25, 2019. doi: 10.1007/s002170050449.
- LAL, R. The role of residues management in sustainable agricultural systems. **Journal of Sustainable Agriculture**, v.5, n.4, p.51–78, 2008. Available from: <[https://doi.org/10.1300/J064v05n04\\_06](https://doi.org/10.1300/J064v05n04_06)>. Accessed: Aug. 27, 2021. doi: 10.1300/J064v05n04\_06.
- MALAVOLTA, E. et al. **Avaliação do estado nutricional das plantas: princípios e aplicações**. 2.ed. Piracicaba: Associação Brasileira para Pesquisa da Potassa e do Fosfato, 1997.
- PADUIT, N. et al. Nutrient uptake and distribution in black pepper. **Better Crops with Plant Food**, v.102, n.4, p.24–27, 2018. Available from: <<https://doi.org/10.24047/BC102424>>. Accessed: Apr. 15, 2019. doi: 10.24047/BC102424.
- PANDEY, R. Mineral nutrition of plants. In: BAHADUR, B. et al. **Plant Biology and Biotechnology**. New Delhi: Springer India, 2015, p.499–538.
- PARTELLI, F. L. Nutrition of black pepper (*Piper nigrum* L.) - a Brazilian experience. **Journal of Spices and Aromatic Crops**, 2009. v.18, n.2, p.73–83. Available from: <<https://updatepublishing.com/journal/index.php/josac/article/view/4942>>. Accessed: Nov. 15, 2019.
- PLATEL, K. & SRINIVASAN, K. Bioavailability of micronutrients from plant foods: An update. **Critical Reviews in Food Science and Nutrition**, 2016. v.56, n.10, p.1608–1619. Available from: <<https://doi.org/10.1080/10408398.2013.781011>>. Accessed: Aug. 25, 2021. doi: 10.1080/10408398.2013.781011.
- PRADEEP, K. U. et al. Common Indian spices: Nutrient composition, consumption and contribution to dietary value. **Plant Foods for Human Nutrition**, v.44, n.2, p.137–148, 1993. Available from: <<https://doi.org/10.1007/BF01088378>>. Accessed: Apr. 15, 2021. doi: 10.1007/BF01088378.
- PREZOTTI, L. C. et al. **Liming and fertilization recommendation for the State of Espírito Santo, Brazil**. 5.ed. Vitória: SEEA - Incaper - CEDAGRO, 2007.
- RAVINDRAN, P. N. **Black pepper: *Piper nigrum***. Kozhikode: Medicinal & Aromatic Plants Industrial Profiles, 2003.
- SALEH-E-IN, M. M. et al. Chemical composition and pharmacological significance of *Anethum Sowa* L. Root. **BMC Complementary and Alternative Medicine**, v.17, n.1, p.127, 2017. Available from: <<https://doi.org/10.1186/s12906-017-1601-y>>. Accessed: Apr. 15, 2021. doi: 10.1186/s12906-017-1601-y.
- SCHILS, R. et al. Fertilizer replacement value: linking organic residues to mineral fertilizers. In: MEERS, E. et al. **Biorefinery of inorganics: recovering mineral nutrients from biomass and organic waste**. Croydon: John Wiley & Sons Ltd, 2020, p.189–214, 2020. Available from: <<https://doi.org/10.1002/9781118921487.ch5-1>>. Accessed: Aug. 27, 2021. doi: 10.1002/9781118921487.ch5-1.
- SIVARAMAN, K. et al. Agronomy of black pepper (*Piper nigrum* L.) - a review. **Journal of Spices and Aromatic Crops**, v.8, n.1, p.1–18, 1999. Available from: <<https://updatepublishing.com/journal/index.php/josac/article/view/4508>>. Accessed: Feb. 27, 2019.
- SRINIVASAN, V. et al. **Nutrient management in black pepper (*Piper nigrum* L.)**. Wallingford: CAB Reviews - Perspectives in Agriculture, Veterinary Science, Nutrition and Natural Resources, 2008. Available from: <<http://dx.doi.org/10.1079/PAVSNNR20072062>>. Accessed: Aug. 27, 2021. doi: 10.1079/PAVSNNR20072062.
- SUPALKOVA, V. et al. Study of capsaicin content in various parts of pepper fruit by liquid chromatography with electrochemical detection. **Acta Chimica Slovenica**, v.54, p.55–59, 2007. Available from: <<http://acta-arhiv.chem-soc.si/54/54-1-55.pdf>>. Accessed: Sep. 9, 2019.
- TAIZ, L. et al. **Plant physiology and development**. 6.ed. Sunderland: Sinauer Associates Inc., 2014.
- TEIXEIRA, P. C. et al. **Methods for soil analysis**. 3.ed. Brasília: Embrapa Solos, 2017.
- VELOSO, C. A. C. & CARVALHO, E. J. M. Nutrient uptake and extraction by black pepper cultivar “guajarina”. **Scientia Agricola**, v.56, n.2, p.443–447, 1999. Available from: <<https://doi.org/10.1590/S0103-90161999000200026>>. Accessed: Oct. 15, 2019. doi: 10.1590/S0103-90161999000200026.
- VIÉGAS, I. J. M. et al. Mineral composition and visual symptoms of nutrients deficiencies in long pepper plants (*Piper hispidinervum* C. DC.). **Acta Amazonica**, v.43, n.1, p.43–50, 2013. Available from: <<https://doi.org/10.1590/S0044-59672013000100006>>. Accessed: Oct. 29, 2019. doi: 10.1590/S0044-59672013000100006.
- YU, Y. et al. Combination of agricultural waste compost and biofertilizer improves yield and enhances the sustainability of a pepper field. **Journal of Plant Nutrition and Soil Science**, v.182, n.4, p.560–569, 2019. Available from: <<https://doi.org/10.1002/jpln.201800223>>. Accessed: Aug. 27, 2021. doi: 10.1002/jpln.201800223.