An approach into the detection of authenticity of black pepper (*Piper nigrum* L.) oleoresin

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ABSTRACT

Investigations on authenticity of black pepper (*Piper nigrum*) oleoresin indicated that saponification value, iodine value and ratio of saponification value to iodine value are useful indicators. The presence of papaya seeds in black pepper used to prepare the oleoresin could be detected at 15 per cent levels using these indices. The advantages of these indices over piperine as an indicator are discussed.

Key words : adulteration, black pepper, oleoresin, piperine, *Piper nigrum.*

Adulteration of black pepper (*Piper nigrum* L.) and its products have been reported from time to time (Bhalla & Punekar 1975; Archer 1987). The major types of adulteration include use of oil or oleoresin extracted seeds, addition of foreign matter in the form of filth, addition of seeds resembling black pepper and addition of starch to powdered samples of black pepper.

Papaya seeds (*Carica papaya* L.) resemble black pepper and are its major adulterant (Bhalla & Punekar 1975). Techniques based on presence of benzyl isothiocyanate in papaya seeds (Chan & Heu 1978; Curl & Fenwick 1983), chromatographic behaviour and UV characteristics (Hartman, Divakar & Rao 1973), staining tests based on presence of starch in black pepper and medium length dextrin in papaya seeds (Shreedharan, Mangalakumari & Mathew 1981) and alcohol floatation tests (Pruthi & Kulkarni 1969) are used to detect papaya seeds in whole black pepper. However no method is reported that can detect whether the black pepper oleoresin is obtained from genuine unadulterated black pepper or from those adulterated with papaya seeds. This work reports on this problem by making use of oil characteristics of black pepper oleoresin and papaya seed oil.

Whole black pepper (Panniyur -1 variety) was obtained from Konkan Agricultural University, Dapoli, India and papaya seeds (Australian variety), procured from a reliable source were selected for the present study. Model

blends of black pepper : papaya seeds in the proportion 100 : 0, 90 : 10, 80 : 20, 70 : 30, 60 : 40, 50 : 50 and 0 : 100 respectively, were prepared and ground to 150 mesh sieve. Oleoresins from these samples were prepared by extracting with ethanol using soxhlet apparatus. The extract was evaporated on a steam bath and heated at 30°C in an oven for 30 min, cooled and weighed (Mathai 1988). The oleoresins so obtained were analysed for saponification value and iodine value as per standard procedures (AOAC 1984). Piperine content in the oleoresin samples was estimated spectrophotometrically at 343 nm using a standard graph prepared from piperine in the range of 2.5 to 10.0 µg/ml. The solvent used in the estimation was benzene (Fagen, Kolen & Hussong 1955; Genest, Smith & Chapman 1963).

The saponification value and iodine value of oleoresins obtained from genuine black pepper and that adulterated with varying levels of papaya seeds are given in Table 1. Papaya seed is known to contain fixed oil in the range of 25.29 to 32.97 % having iodine values ranging from 72.60 to 74.77, and saponification value ranging from 189.5 to 193.4 (Loesecke & Nolte 1937; Chan & Heu 1978). Black pepper on the other hand contains both fixed oil (6.0 - 10.0%) and oleoresin (about 9%) (Krishnamurthy 1969).

Iodine value has been suggested as a means for detecting adulteration in essential oils (Kartha & Mishra 1963; Kumar & Madaan 1970). Edible and mineral oils have been used for adulterating essential oils and physical methods such as ester number, solubility, specific gravity and optical rotation have been used for detecting such malpractices (Mostafa, Gomaa & El-Masryh 1990). The wide differences in saponification value and iodine value of pure oleoresin from black pepper and oil from papaya seeds suggests them to be useful quality criteria for checking the authenticity of black pepper oleoresin.

Table 1. Saponification value and iodine value of oleoresins of admixtured samples of black pepper powder and papaya seeds

Blend of black pepper : papaya seeds	Saponification value (A)	Iodine value (B)	A/B
100 : 0	72.7 ± 1.50	104.3 ± 5.5	0.697
95 : 5	80.4 ± 1.00	100.1 ± 3.1	0.803
90 : 10	95.0 ± 1.75	96.1 ± 3.0	0.990
85 : 15	105.0 ± 1.30	92.0 ± 2.1	1.140
80 : 20	117.6 ± 1.35	87.4 ± 1.2	1.346
70 : 30	ND	83.1 ± 1.5	ND
0 : 100	194.2 ± 2.00	70.3 ± 2.5	2.760

Results are mean ± SD of three individual determinations
ND = Not determined

The ratio of saponification value to iodine value is also promising. The ratio changed rapidly from 0.697 for pure black pepper oleoresin to 1.140 in oleoresin prepared from black pepper adulterated with as low as 15 per cent papaya seeds. Piperine content is also useful in detecting this blending, but there are several considerations which should be taken into account in using this as an indicator of black pepper oleoresin. Piperine content is unique to black pepper rather than the adulterant; its content could vary from variety to variety, cultivar to cultivar and could also be sensitive to season and geographical origin; its volatile nature and its sensitivity to light are other factors which prevent it from being used as a quality parameter to check the authenticity of black pepper oleoresin. However, it could be used, provided the sample is analysed within a short time of manufacture (Table 2) and a pure authentic sample of the spice used in the manufacture of oleoresin is available for comparative analysis.

Table 2. Piperine content of oleoresins of admixtured samples of black pepper powder and papaya seed powder

Blend of black pepper : papaya seeds	Piperine in oleoresin (%)
100 : 0	47.16
90 : 10	42.34
80 : 20	37.45
70 : 30	31.63
50 : 50	23.72

Results are means of three individual determinations

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