JETIR.ORG ISSN: 2349-5162 | ESTD Year : 2014 | Monthly Issue
JOURNAL OF EMERGING TECHNOLOGIES AND INNOVATIVE RESEARCH (JETIR)
An International Scholarly Open Access, Peer-reviewed, Refereed Journal

Extraction, Estimation and Characterization of Peperine from Black Pepper

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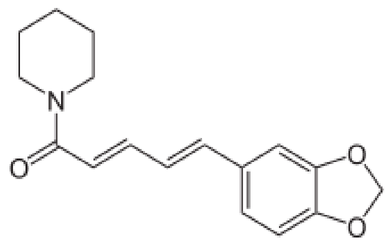
Abstract
Black pepper (Piper nigrum) belongs to the Piperaceae family. It contains Piperine, an active ingredient which is a Nitrogen-containing alkaloids. It's a spices crop, which is used as to enhancing the digestion of food and an essential component in commercial medicine. In this study we report the extraction of Piperine from black pepper and characterize it using XRD methods.

Keywords: Black pepper, Piperine, XRD.

Introduction

Pepper is often described as the "King of spices" and it share a place on most dinner tables with salt. The word pepper is originated from the Sanskrit word 'Pippali,' which means berry. A pepper is a pungent hot-tasting powder prepared from dried and ground peppercorns, used as a spice or condiment to flavor food [1]. It is a vegetable with variable shape, size, and color. It can be green, red, white, and even black. Its taste can be sweet or spicy and it can be consumed fresh, stewed, preserved, and can be used as a garnish in a great variety of dishes. The hot taste sensation in pepper comes from a resin called chavicine in the peppercorns. Peppercorns are also the source of other heat-generating substances, including an alkaloid called Piperine, which is used to add the pungent effect in varieties of dishes. Piperine along with its isomer chavicine, is an organic compound that falls under the lipid family, which is made up of nitrogenous compounds with physiological qualities like fat or substances that resembles fat. This is among the components of fruits with a pungent flavor, such as long pepper (Piper Longum) and black pepper (Piper Nirgun)[1,2,3]. The existence of the isomer chavicine, which shares the same chemical formula but a distinct molecular structure from Piperine, accounts for the pepper's strong flavor. Long-term storage of peppers causes their pungency of fade because chavicine slowly transforms into Piperine[4,5]. Both black and white pepper come from the shrub classified as 'Piper nigrum' which is one of about thousand species the Piper genus that is part of the larger family of peppers called Piperaceae. The percentage of Piperine present in black pepper is around 2.5% - 3% and in white pepper is around 4% - 4.5%.



The molecular formula of Piperine is $C_{17}H_{19}NO_3$ and it belongs to the family of alkaloids. It is an organic nitrogenous chemical with a planar molecular structure. A carbon-carbon double bond connects the aromatic phenyl ring to the Piperidine ring in the molecular structure.

Theoretical Background

The Soxhlet extraction method is relatively economical and only requires a tiny volume of solvent. The Soxhlet extraction method [6] preserves solvent extraction efficiency and achieves high efficiency by continually extracting solid materials using a pure solvent and the solvent reflux and siphon principle. The Soxhlet extractor is built after the solid sample is put on a filter paper that resembles a thimble. The solvent reservoir flask is filled with solvent and placed on a heating mantle. Following heating, the sample powder and the solvent's condensed vapours come into contact, mixing the soluble portion of the powder with the solvent in preparation for extraction. The solvent holding the extract is siphoned back when the solvent surface rises above the siphon's maximum height. To ensure that the solid material is always utilized as a pure solvent and that the extracted material is concentrated in the flask, the flask is reused, extracting a portion of the substance each time.

Experimental

All chemical used were of AR grade. The XRD analysis is done ousing Rigaku Smart Lab X-Ray Diffractometry (CuK- α radiation 1.5402 A^0).

Extraction of Piperine: Requisite amount of Black Pepper is grinded to a coarse powder. It was then treated with 99% Isopropanol in a Soxhlet extraction apparatus for 2 hours. The solution was allowed to cool down and filter. It was then concentrated and the product extracted using 10% alcoholic KOH from the residue. The solution is then cooled to get the product as precipitate. The product is purified by recrystallization using isopropanol. Yellow crystalline product is collected (yield 20.4 %; mp 125 ˚C) [1,2].

Result and Discussion

X-Ray Diffraction Studies: Using the XRD pattern obtained from the Rigaku Smart Lab X-Ray Diffractometry (CuK- α radiation 1.5402A^0), the phase purity and crystallite nature of the Piperine were examined. Figure 1 displays the Piperine powder's XRD pattern. Sharp peaks are seen in Piperine powder, indicating that it has a crystalline structure. The main diffraction peaks of Piperine occurs at 2Θ = 14.74, 19.64, 22.35, 25.73 and 28.12. The sharp peaks and their relative intensities are listed in Table 1. The size of the Piperine complex calculated by the Debay Scherrer Equation is in the range of 2.44 nm.

Debay Scherrer Equation $\mathbf{D = K\lambda / \beta \cos\theta}$

$$D_{XRD} = 0.98\lambda/\beta cos\theta$$

λ= Wavelength of x-ray (0.154nm)

β= Full width at half- maximum in degree in the 2θ scale

θ = Bragg angle

$$\mathbf{D_{XRD} = \frac{0.15092}{0.06163} = 2.44nm}$$

Table 1 measured XRD peak position and relative intensities of Piperine powder.

Peak position (2Θ)	Relative intensities (cps)
14.76	208480
19.67	100770
22.29	194540
25.71	226270
28.14	68567

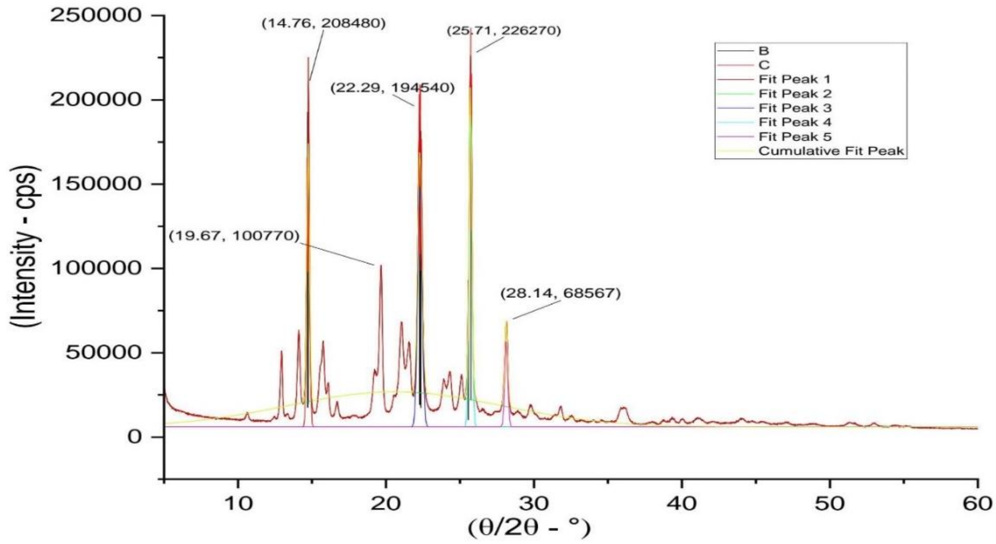


Fig 1 - XRD pattern with peak positions of Piperine powder

Conclusion

The isolated Piperine alkaloids from black pepper seeds were characterized by melting point 124˚C which showed that it undergoes melting at 124˚C for black pepper which is close to the standard range (129˚C - 130˚C). Also from the XRD analysis, sharp peaks have been seen in Piperine powder, indicating that it has a crystalline structure and the main diffraction peaks of Piperine occurs at 2Θ= 14.74, 19.64, 22.35, 25.73 and 28.12.

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