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Chemometric Resolution for Baseline Identification of *Amomum subulatum* Roxb. Essential Oils through FT-IR Spectroscopy Technique

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Abstract

Essential oil of *Amomum subulatum* is the complex mixture of various alkyl, alkanes, sulfonates, amines, alcohols, sulfoxides, *trans* RCH≡CHR and aromatic bending compounds. The FT-IR analysis of aromatic bending occurs on 845 cm^{-1} with *para* C-H bend. A strong absorption band between 900 cm^{-1} - 675 cm^{-1} indicated the presence of aromatic C=C. The alcoholic stretching detected on 1080 cm^{-1} and 1169 cm^{-1} with C-O stretching molecular motion in large cardamom. In our investigation, the frequency of 2943 cm^{-1} and 2934 cm^{-1} the C-H stretching occurred in alkyl group. The wave numbers of bands 2966 ($C=CH_2$), 1358 (S=O Stretch), 1215 (C-N Stretch), 1053 (S=O Stretch) and 984 (C-H Bending) the functional group specify vinyl, sulfonates, amines, sulfoxides and *trans* RCH-CHR, respectively.

Keywords

Essential Oil, 1,8 Cineole, FTIR, Spectra

1. Introduction

Amomum subulatum Roxb. belongs to the family Zingiberaceae is well adopted to cultivation in the hilly areas of Uttarakhand, India [1] and other states such as Sikkim, West Bengal [2]. It is mainly cultivated as spice however, also possesses several medicinal properties such as carminative, stomachic, diuretic and cardiac stimulant [1]. The essential oil yield was reported to be between 2.5% to 3% for

the cardamom grown in different agro-climatic regions of Uttarakhand [1], which contains 31 compounds with 1,8 Cineole as major compound.

Terpenoids, aldehydes, ketones, alcohols and esters were the main compounds present in essential oil. Several methods have been developed for the isolation and identification of compounds present in the essential oil such as Supercritical Carbon Dioxide Extraction (SCDE), Gas Chromatography Mass Spectrometer (GC-MS) and Fourier Transform Infra Red Spectroscopy (FT-IR). FT-IT is a technique through which the functional group presents was determined through comparing the vibration frequencies in wave numbers of the sample spectrograph with those of the IR correlation chart [3]. Identification through FT-IR technique is an advanced chemometric resolution technique through which primary identification of compounds present in essential oil has become possible. Furthermore, this technique is also used for the analysis of unresolved peaks using multivariate curve resolution [4].

Keeping this in view, the present study was designed to identify the presence of 1,8 Cineole in the *A. subulatum* essential oil grown in different agro-climatic regions of Uttarakhand, India. This is the base-line information on presence of 1,8 Cineole in *A. subulatum* essential oil.

2. Material and Methods

2.1. Collection and Identification of Plant Materials

The capsule of the large cardamom was collected during the month of October-November from different agro-climatic regions of Uttarakhand viz. Guhad (Chamoli), Kwiti (Pithoragarh), Lamgarha (Almora), Mandal (Chamoli), Parkandi (Rudraprayag), Sema (Tehri) and Singot (Uttarakashi). The plant was identified by Botanical Survey of India, Dehradun, India with the Accession number 117,262.

2.2. Oil Extraction

The dried whole capsules were subjected to hydro-distillation for extraction of essentials oils using Clevenger apparatus for 6 hours at a temperature of 90˚C. The collected concentrated oil was then subjected to anhydrous sodium sulphate to remove the moisture contents and then stored at 4˚C for further analysis.

2.3. Standard Solution

Pure standard compound of 1,8-cineole (MF $C_{10}H_{18}O$, MW 154.3 g/mol, Assay 98%) was purchased from HWI Analytik, Germany.

2.4. Fourier Transforms Infrared Spectroscopy (FT-IR) Technique

The ATR-FT-IR spectra were recorded in a range between 8500 - 485 cm^{-1} using a FT-IR (Make: Horizon MB, spectrometer and detector; Model 114,690 - 131,082, TGS, apodization Cosine, Make-Horizon MS and S.No-1405772-001). Essential oil 5 - 10 ml was placed on the surface of the diamond ATR crystal. The spectral

data were processed with Horizon MB Spectra software. Samples were scanned at 4 cm^{-1} resolution, accumulation: 100 scans.

3. Results and Discussions

The structure of 1,8 Cineole is provided in Figure 1. The FT-IR analysis of the essential oil of *A. subulatum* revealed the presence of different functional group such as, alkyl, vinyl, alkanes, sulfonates, amines, alcohol and sulfoxides (Table 1, Figure 2). The functional groups present in essential oil were determined through evaluating the vibration frequencies in wave numbers of the sample spectrograph obtained from FT-IR spectrophotometer with those of a correlation with IR table. The vibration frequencies of the solvent were also obtained to aid in the determination of sample vibration frequencies. The saturated hydrocarbon C-H stretching absorption occurs below 3000 cm^{-1}. The alcoholic stretching detected on 1080 cm^{-1} and 1169 cm^{-1} with C-O stretching molecular motion in large cardamom. The position of the C=C stretching frequency does vary

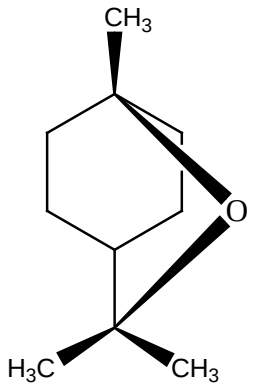

Figure 1. Structure of 1,8 Cineole.

Table 1. Wave number of band with molecular motion in vibration assignments for oils essential oils of *A. subulatum* (Roxb.)

Wave number of bands (cm^{-1})	Molecular motion	Functional group
2966	$C=CH_2$	Vinyl
2943	C-H Stretch	Alkyl
2924	C-H Stretch	Alkyl
1377	CH_3 Bend	Alkanes
1358	S=O Stretch	Sulfonates
1215	C-N Stretch	Amines
1169	C-O Strong	Tertiary alcohol
1080	C-O Stretch	Primary alcohol
1053	S=O Stretch	Sulfoxides
984	C-H Bending	trans RCH=CHR
845	C-H Bend (para)	Aromatic Bending

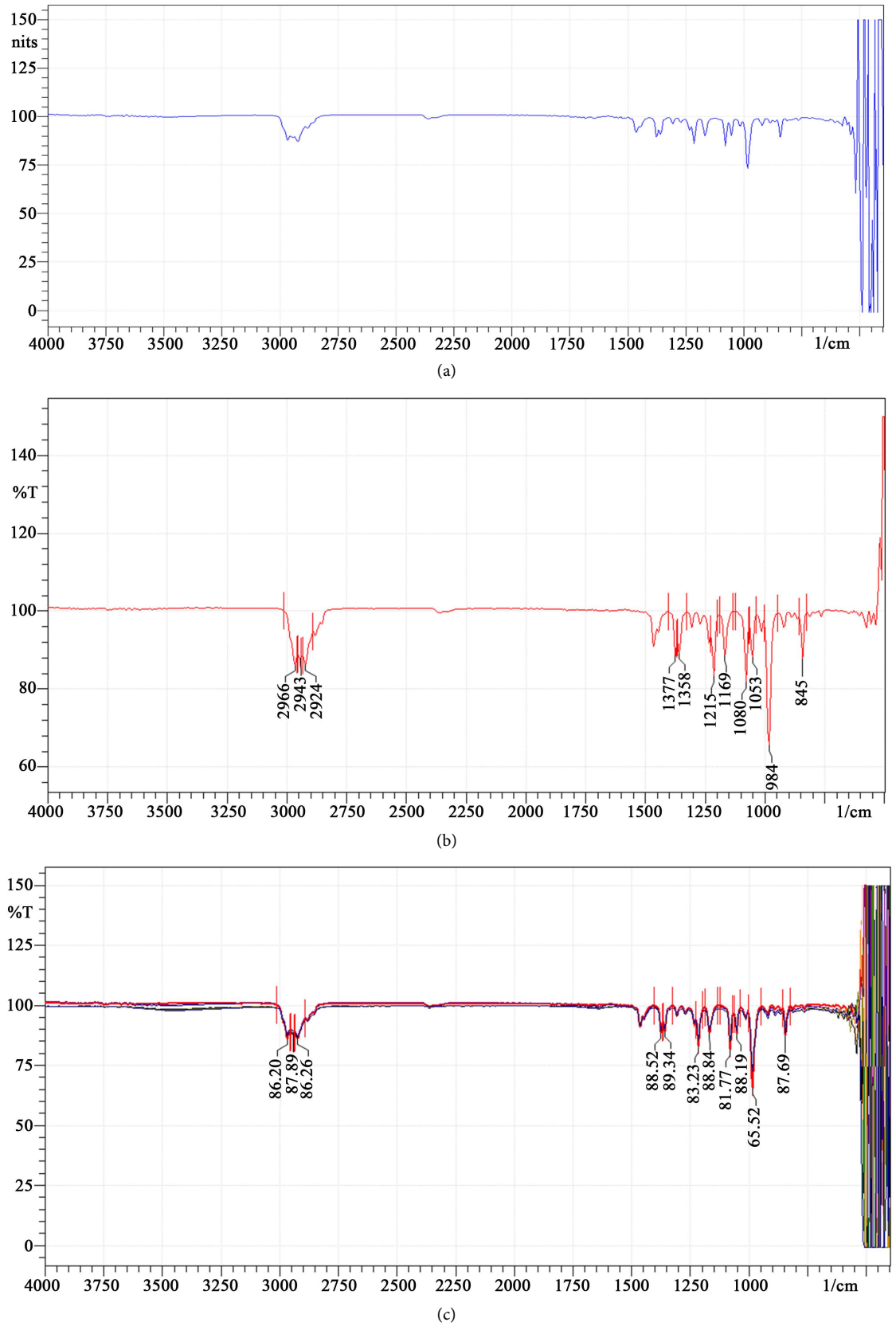

Figure 2. FT-IR spectra of *A. subulatum*: (a) Spectra of *A. subulatum* oil sample; (b) 1,8 cineole spectra; (c) Overlay spectra of 1,8 cineole and *A. subulatum* oil sample of different sites.

slightly as a function of orientation around the double bond, but it is less informative than the C–H information. Aromatic bending occurs on 845 cm^{-1} with para CH bend. A strong absorption band between 900 cm^{-1} to 675 cm^{-1} indicated the presence of aromatic C=C. The absorbance band at 1377 cm^{-1} revealed the presence of CH_3 bond for alkanes. From the spectra we can see clearly that although they show substantial overlap of each absorption spectrum of various components, each band represents an overall overlap of some characteristic absorption peaks of functional groups in the samples. Therefore, FT-IR spectrum reflecting objectively the panorama of chemical constituents in complex system is a most credible method to validate and identify the mix-substance systems such as traditional medicine and herbal medicine [5]. It is observed in present study that the C-H stretching occurred in alkyl group between the frequency of 2943 cm^{-1} and 2934 cm^{-1}. The wave numbers of bands 2966 ($C=CH_2$), 1358 (S=O Stretch), 1215 (C-N Stretch), 1053 (S=O Stretch) and 984 (C-H Bending) the functional group occurs Vinyl, sulfonates, Amines, sulfoxides and *trans* RCH ≡CHR found respectively (**Table 1**, **Figure 2**). The result of the FT-IR analysis of essential oil of *A. subulatum* of seven different sites were found relatively identical for the 1,8 cineole compound (**Figure 1**). Earlier studies on essential oil of *A. subulatum* also revealed the presence of 1,8 cineole as a major compound [1] [5]. Many workers applied the FT-IR spectrum as a tool for differentiating, classifying and discriminating closely related plants and other organisms [6] [7]. Thus, finding of the present work on *A. subulatum* oils is used as analytical tool to check not only the primary identification of the compounds present in oils but also help in to further examine those compounds for their biological properties.

4. Conclusion

The results of the present study revealed the presence of 1,8-cineole in all the essential oils analyzed. Vinyl, alkyl, alkanes, sulfonates, amines, alcohols, sulfoxides, *trans* RCH≡CHR are the other important functional groups identified in the essential oil of *A. subulatum* grown in different agro-climatic regions of Uttarakhand, India.

References

[1] Bhandari, A.K., Baunthiyal, M., Bisht, V.K., Negi, B.S. and Kaim, J.C. (2017) Assessment of Intra-Specific Variation in Essential Oil Composition in *Amomum subulatum* Roxb. Cultivated in Uttarakhand, India. *Asian Journal of Chemistry and Pharmaceutical Sciences*, **2**, 1-5.

[2] Balakrishnan, K.V., George, K.M., Mathulla, T., Pillai, Narayana, O.G., Chandran, C.V. and Verghese, J. (1984) Studies in Cardamom-1, Focus on Oil of *Amomum subulatum* Roxb. *Indian Spices*, **21**, 9-12.

[3] Madivoli, S.E., Gitu, L. and Gumba, E. (2012) Isolation and Identification of Essential Oils from *Cymbopogan citrates* Dc Using GC-MS and FT-IR. *Chemistry and Materials Research*, **2**, 13-22.

[4] Heravi-Jalali, M., Moazeni Sadat, R. and Sereshti, H. (2011) Analysis of Iranian Ro-

semary Essential Oil: Application of Gas Chromatography—Mass Spectrometry Combined with Chemometrics. *Journal of Chromatography A*, **1218**, 2569-2576. https://doi.org/10.1016/j.chroma.2011.02.048

[5] Bhandari, A.K., Bisht, V.K., Negi, J.S. and Baunthiyal, M. (2013) 1,8-Cineole: A Predominant Component in the Essential Oil of Large Cardamom (*Amomum subulatum* Roxb.). *Journal of Medicinal Plants Research*, **7**, 1957-1960.

[6] Liu, H., Sun, S., Lv, G. and Chan, K.K.C. (2006) Study on Angelica and Its Different Extracts by Fourier Transform Infrared Spectroscopy and Two-Dimensional Correlation IR Spectroscopy. *Spectrochimica Acta Part A*, **64**, 321-326. https://doi.org/10.1016/j.saa.2005.07.026

[7] Lamprell, H., Mazerolles, G., Kodjo, A., Chamba, J.F., Noel, Y. and Beuvier, E., (2006) Discrimination of *Staphylococcus aureus* Strains from Different Species of *Staphylococcus* Using Fourier Transform Infrared (FTIR) Spectroscopy. *International Journal of Food Microbiology*, **108**, 125-129. https://doi.org/10.1016/j.ijfoodmicro.2005.10.015