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ORIGINAL

Application of headspace solid-phase microextraction gas chromatography mass spectrometry (HS-SPME GC-MS) analysis to support illicit timber trafficking investigations

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Abstract

Large quantities of timber are covertly trafficked through legitimate maritime routes, however the complexity of global trade networks, combined with inadequate detection tools, enables trafficking to persist and hinders prosecution efforts. This study investigates the feasibility of volatilome profiling using Gas Chromatography Mass Spectrometry (GC-MS) for the detection and identification of *Pterocarpus indicus*, a commercially and ecologically significant species, via air samples. Headspace (HS) analysis was performed using solid-phase microextraction (SPME) with samples subjected to different treatments (untreated, oven drying or freeze drying), or mixed in different ratios with pine (*Pinus radiata*), a common legally exported wood. Sixteen compounds were tentatively identified from *P. indicus*, predominantly consisting of oxygenated sesquiterpenes, the emission of which was adversely influenced by drying methods. The volatilomes profiled from each wood type could also be visually differentiated in mixed samples and form the basis of a preliminary screening approach for identifying high-risk timber species in heterogenous timber shipments. Our preliminary findings suggest that HS-SPME GC-MS could be adapted as a non-invasive screening approach for cargo container inspections, facilitating initial assessment of potential trafficking activity without compromising cargo integrity or necessitating intrusive inspection procedures.

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Introduction

Illegal logging operations have devastated forests globally, resulting in severe biodiversity loss and habitat destruction (Lin et al. 2021; Pan et al. 2024). With an estimated 15–30% of all timber traded globally being sourced illegally, these operations have also contributed to substantial economic losses and pronounced environmental consequences (INTERPOL 2021). Rising demand for timber intensifies pressure on already degraded forests and protected areas, as financial incentives drive organised criminal groups to engage in illegal logging (UNODC 2025). Environmental crime is facilitated by corruption, inadequate law enforcement and the challenges associated with monitoring its infiltration into legitimate global supply chains, making effective enforcement difficult (UNODC 2025).

Rosewood is currently the most valuable taxa traded globally, driven by high demand in international markets (Dumenu 2019; Nijman 2024; Siriwat and Nijman 2023). Rosewood, a term used to describe a specific group of high-quality tropical hardwoods known for their rich colour and fine grain, is highly sought after for producing luxury furniture, musical instruments and decorative items (Zhang et al. 2022a, b; Zhu 2022). Despite international efforts, such as the protection of many rosewood species under the Convention on International Trade in Endangered Species (CITES) (CITES Secretariat 2024), illegal trade persists, underscoring the need for strengthened regulatory frameworks, sustainable forestry practices and greater enforcement to combat forestry crime (UNODC 2025).

Pterocarpus indicus, commonly known as 'narra', is a hardwood native to the Asia-Pacific region (Winfield et al. 2016). While it is not a true 'rosewood' (a term normally reserved for *Dalbergia* spp.), it is within the same Family (*Fabaceae*) and shares many similarities. It is thus considered one of the 29 species included within the Hongmu ('Hongmu' translates to 'red wood') National Standard of the People's Republic of China (GB/T 18107–2017), and is subsequently highly valued in the Chinese market for high-end furniture (National Standard of the People's Republic of China 2017; Zhang et al. 2022a, b). The primary threat to this species is illegal logging and it currently holds an endangered International Union for Conservation of Nature (IUCN) rating, with native populations declining (Barstow 2018). African populations of *Pterocarpus* spp. were recently identified as being at risk due to historical and continued overexploitation for timber and have consequently been added to CITES Appendix II (CITES 2022) - as *P. indicus* is also considered endangered by trade, officials have considered adding this species in Appendix II before they become commercially extinct (Rutherford et al. 2023). This species has already been identified as a priority species in some regions with legislative restrictions surrounding its use (Winfield et al. 2016). Such protections must be implemented proactively, otherwise, as seen in the case of *P. erinaceus*, populations may become commercially extinct by the time CITES regulations are enforced, undermining their effectiveness (UNODC 2024).

Despite legislative protections, rosewood continues to be harvested and traded illegally. Notable trends include large-scale seizures at major seaports as traffickers attempt to smuggle rosewood in shipments disguised as legal timber or other goods (UNODC 2024). Timber can in itself also be used to conceal other illicit products such

as wildlife and frequently converge with broader illicit activities (UNODC 2025). As traffickers adapt, the need for continued international collaboration and advanced forensic tools remains critical to addressing the illegal rosewood trade. In particular, there is an absence of detection tools available to non-invasively discern the contents of cargo containers, especially for discriminating between different types of wood, without needing to open the container. Current operations rely on intelligence and risk-assessments, searching for risk indicators such as fraudulent documentation or weight discrepancies (Zavagli 2021), however this does not constitute definitive evidence of the presence of an illicit species within the container. Confirmatory approaches, including manual inspections, can be costly, time-consuming and may be ineffective, particularly when illegal timber is concealed behind legally sourced wood, without comprehensive screening and complementary verification methods.

Best practices for the forensic identification of timber species have been established and include various approaches based on visual chemical and genetic features (UNODC 2016). Commonly used techniques include morphological analysis (i.e. XyloTron; Ravindran et al. 2020), DNA barcoding (But et al. 2023; Dev et al. 2023) and chemical fingerprinting (i.e. via Direct Analysis in Real Time Time-of-Flight Mass Spectrometry (DART-TOFMS); Isak et al. 2025; Lancaster and Espinoza 2012; Price et al. 2021), though these techniques typically require physical samples which may limit inspection approaches. Volatilome profiling has emerged as a promising method for both detection and species determination, which may offer an effective solution to both deciphering the presence of illicit wood and discerning the identification of rosewood products, or alternatively to recognise counterfeits. Gas Chromatography-Mass Spectrometry (GC-MS) is a versatile technique which has been successfully applied to distinguish *Pterocarpus* species from extracts and headspace analysis, as well as other wildlife derivatives (Moloney et al. 2026a; Ueland et al. 2016, 2020), for illicit trade investigations (Zhang et al. 2019, 2022a, b) and warrants further exploration. Analysis of the headspace could offer particular advantages for trade investigations, especially when timber is concealed within containerised cargo, as volatile compounds equilibrate in an enclosed space. This approach could facilitate non-invasive and non-destructive screening of shipments to detect misdeclared or undeclared wood types, providing a rapid preliminary assessment without compromising cargo integrity.

Our study aimed to determine whether reproducible and distinguishable volatilomes could be detected in the headspace of *Pterocarpus indicus*, an ecologically and commercially significant species. The volatilome profile derived and compounds tentatively identified were subsequently applied to a small-scale mixed-wood scenario to assess whether *P. indicus* could be distinguished in the presence of another common timber species (*Pinus radiata*), simulating a plausible concealment scenario. The overarching objective was to assess the feasibility of generating comparative and distinguishable volatilome profiles, rather than to define definitive species-specific chemical biomarkers, with the aim of informing exploratory air-sampling approaches in trade monitoring contexts.

Methods

Sample preparation

P. indicus samples ($n=2$) were obtained from two different commercial sources in Adelaide, Australia. Pieces of *P. indicus* wood were either left untreated, or were prepared using one of two commonly employed preservation methods for comparison: oven-drying (at 50 °C for seven days) or freeze-drying. Fresh radiata pine (*Pinus radiata*) was also collected from a local plantation and remained untreated. All wood samples (untreated and treated) were cut into smaller pieces and then ground to a fine powder using a ball mill at 30 Hz (Mixer Mill MM-400, Retsch, Germany).

Powdered wood samples were placed into airtight 20 mL solid phase microextraction (SPME) glass vials with a screw cap containing a polytetrafluoroethylene/silicone septum (Supelco, USA). Samples consisted of 0.5–1 g (deviation tolerance ± 0.004 mg) of powdered wood, comprising either a single wood type or a mixture of rosewood and pine. For mixed samples, untreated rosewood and pine were combined to a total mass of 1 g in ratios of 0.5:0.5, 0.25:0.75 and 0.1:0.9 (rosewood: pine), respectively, and placed into SPME vials to assess whether they could be reliably distinguished. Pine was added first, and the vial contents were shaken vigorously to ensure thorough mixing. All samples were replicated and analysed in duplicate.

HS-SPME GC-MS analysis

A Supelco 85 μm PDMS/Carboxen SPME was utilised for the extraction and analyses. Prior to analysis, headspace (HS) samples were extracted by inserting and exposing the SPME fibre in SPME vials whilst they were incubated in an oven at 65 °C for 40 min. GC-MS was conducted using a PerkinElmer SQ8 GC-MS (PerkinElmer, USA). The SPME needle was desorbed using splitless injection mode in the injector at 270 °C, with on-column liquid nitrogen cryofocusing in Helium carrier gas at a constant flow rate of 1.2 mL/min. Chromatographic separation was conducted using a 60 m, 0.25 mm ID Agilent DB-5MS capillary (Agilent Technologies, USA) with a temperature program from 50 °C to 300 °C at a ramp of 10 K/min and held for 9 min. Data was collected in election impact mode at 70 eV in fullscan mode from 20 to 400 m/z at approximately three scans per second.

Compound analysis and data visualisation

Data was visualised and interpreted using the TurboMass software (version 6.1.2.2048, PerkinElmer, USA). Peak integration was performed on the total ion chromatogram (TIC) using the automated peak integration function with baseline correction; manual verification was undertaken where necessary to ensure appropriate peak assignment. Total peak area was calculated as the sum of all integrated peaks within a defined retention time (RT) window of 0–30.0 min. Compounds eluting before 4.0 min and compounds contributing less than 0.5% to the relative composition were excluded from further consideration to minimise interference from early-eluting or low-abundance components.

Tentative compound identification was performed by comparison with the National Institute of Standards and Technology (NIST) Mass Spectral Library Database (version 2017). Compound names were assigned with reference to both the literature and NIST 17 where the reverse match score exceeded 700/999. Structural formulae for tentatively identified compounds were generated using ACD/ChemSketch (version 2022.2.3, ACD/Labs, Toronto, Canada). Identifications were considered tentative and were not confirmed using analytical reference standards. Certified reference materials for each reported compound were not available and confirmatory analysis using standards was beyond the scope of this initial proof-of-concept investigation. For mixed samples, the characteristic pattern of target ions corresponding to diagnostic peaks previously identified in the individual sample analyses was evaluated.

Peak areas were used as the analytical signal to represent the relative contribution of each compound. TIC responses within a defined set of chromatograms were normalised to an equivalent scale by expressing individual peak areas as a percentage of the total integrated ion current per chromatogram. Specifically, each peak area was divided by the sum of all integrated peak areas within the TIC and multiplied by 100. This area normalisation approach standardised responses to a common relative scale, enabling comparison between samples independent of absolute signal intensity. TIC data were exported as CSV files using the TurboMass 'peak list write' function and processed in Microsoft Excel for calculation of total peak area and percentage composition. It should be emphasised that the calculated percentage peak areas do not represent the absolute abundance of compounds in the wood samples. Instead, they reflect the relative detector response under the specific HS-SPME GC-MS conditions employed, including analyte affinity for the SPME fibre and the defined extraction parameters. Accordingly, the reported percentages are semi-quantitative and inherently method-dependent and comparisons are valid only within the standardised experimental conditions used in this study.

Results

HS-SPME GC-MS analysis of wood samples

Headspace volatiles were extracted from *P. indicus* and *P. radiata* powdered samples using HS-SPME and analysed by GC-MS, with TICs of representative samples shown in Fig. 1. Negligible differences were profiled between the two sources of *P. indicus*, or their duplicates therein. Most *P. indicus* volatile compounds eluted between 16 and 20 min, with no appreciable compounds identified beyond a RT of 20 min (Fig. 1a, peaks 1–16, tentative compounds listed in Table 1). Conversely, predominant compounds identified in *P. radiata* (Fig. 1b) included alpha-pinene (peak 17, RT 9.11 min, 29% of the total integrated chromatographic area), beta-pinene (peak 18, RT 9.90 min, 27%) and a limonene isomer (peak 19, RT 10.68 min, 18%).

The peak area, calculated by area normalisation of the TIC and represented as a percentage of the total peak area, served as the analytical signal to represent the relative abundance of each compound within a sample under the specified experimental conditions. Compounds with peak areas exceeding 0.5% were tentatively identified

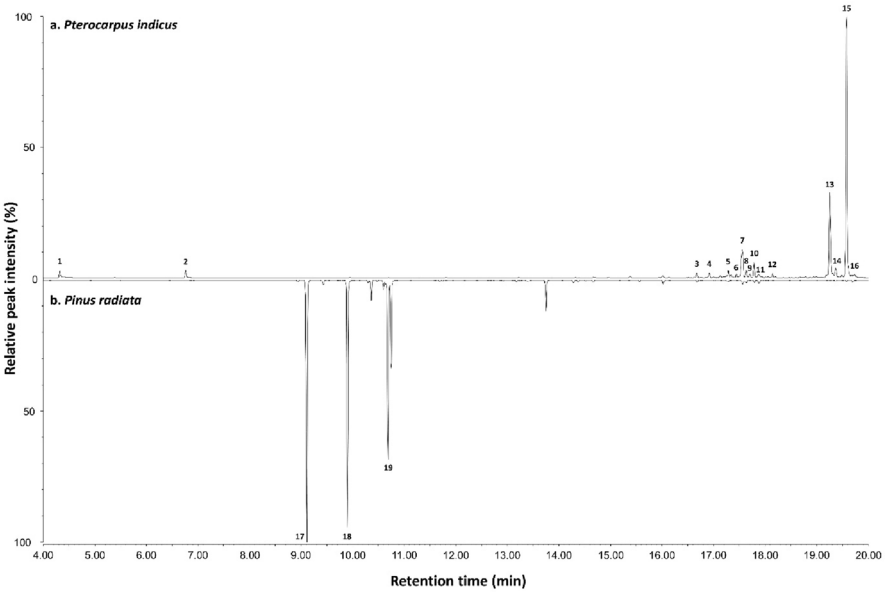


Fig. 1 Mirror plot of representative total ion chromatograms obtained through HS-SPME GC-MS of *Pterocarpus indicus* (1**a**) and *Pinus radiata* (1**b**), including designation of key compounds tentatively identified

by comparing their mass spectra with entries in the NIST 17 Mass Spectral Library Database and relevant literature, as summarised in Table 1. Across the *P. indicus* samples analysed, a total of sixteen distinct volatile compounds were consistently observed, predominately featuring oxygenated sesquiterpenes and sesquiterpene hydrocarbons. The most prominent compounds identified by relative abundance were viridiflorol (peak 15, 47.1% of the total integrated chromatographic area), globulol (peak 13, 17.0%) and an aromadendrene isomer (peak 7, 7.4%).

Influence of sample treatment conditions

P. indicus samples were subsequently analysed following exposure to two treatment conditions: oven-drying (Fig. 2b) and freeze-drying (Fig. 2c). Freeze-drying resulted in the reduced emission of later-eluting volatiles, particularly viridiflorol (peak 15), relative to the untreated sample. The distinction between sample treatments therefore was not in the number of detected molecules identified or compound types emitted, but in the relative concentrations represented by peak area. Meanwhile, oven drying resulted in a reduction in absolute signal intensity, with several peaks (e.g., peaks 13 and 15) showing relative decreases of approximately 50%. This reduction was broadly proportional across detected compounds, such that the overall relative composition of the volatilome remained largely unchanged. Oven drying appeared to have minimal influence on the volatiles emitted beyond a reduction in these same later-eluting compounds and did not reveal any additional compounds compared to those identified in the original trace. Apparent differences in peak visibility are

Table 1 Chemical composition of a representative sample of *Pterocarpus indicus* identified through HS-SPME GC-MS analysis, with tentative compound identification against the NIST 17 Mass Spectral Library Database. Compounds contributing less than 0.5% to the relative composition, or eluting before a retention time (RT) of 4.0 min, are not listed.

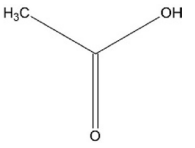
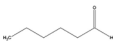
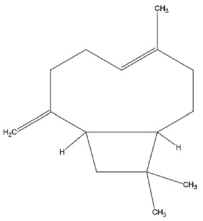
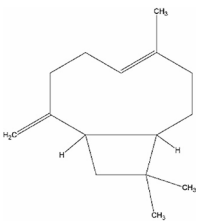
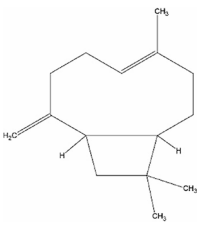
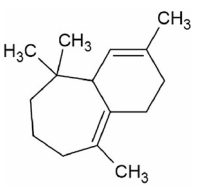
ID	RT (min)	Compound	Structural formulae	Classification	Relative content (%)[1]	NIST reverse match score[2]
1	4.32	Acetic acid		Acid	1.6	900
2	6.76	Hexanal		Aldehyde	1.4	939
3	16.67	Caryophyllene isomer		Sesquiterpene	0.73	900
4	16.92	Caryophyllene isomer		Sesquiterpene	0.91	925
5	17.28	Caryophyllene isomer		Sesquiterpene	1.1	896
6	17.44	Himachalene isomer		Sesquiterpene	0.57	799

Table 1 (continued)

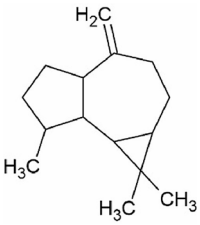
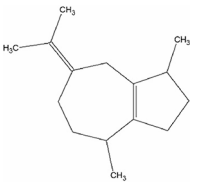
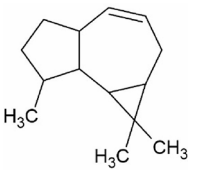
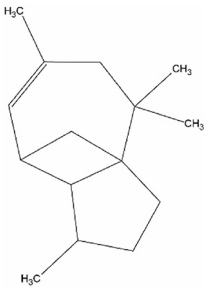
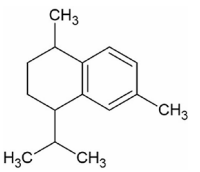
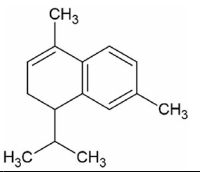
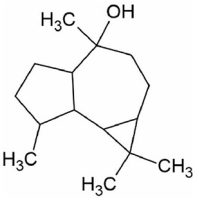
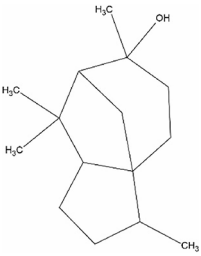
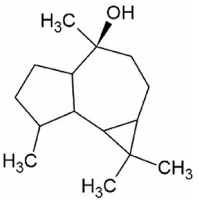
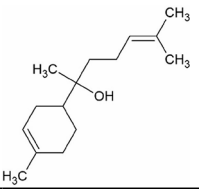
ID	RT (min)	Compound	Structural formulae	Classification	Relative content (%)[1]	NIST reverse match score[2]
7	17.56	Aromadendrene isomer		Sesquiterpene	7.4	959
8	17.63	Guaiene isomer		Sesquiterpene	1.2	906
9	17.70	Aromadendrene isomer		Sesquiterpene	0.63	823
10	17.79	Cedrene isomer		Sesquiterpene	2.5	931
11	17.88	Calamenene isomer		Sesquiterpene	0.84	902
12	18.14	Calacorene isomer		Sesquiterpene	0.62	844

Table 1 (continued)

ID	RT (min)	Compound	Structural formulae	Classification	Relative content (%)[1]	NIST reverse match score[2]
13	19.25	Globulol		Oxygenated sesquiterpene	17.0	982
14	19.37	Cedrol		Oxygenated sesquiterpene	1.6	872
15	19.58	Viridiflorol		Oxygenated sesquiterpene	47.1	994
16	19.73	Bisabolol isomer		Oxygenated sesquiterpene	0.77	889

[1]The percentage was calculated from each peak area relative to the total chromatographic peak area (approx. 2.0×10^8 counts·s), defined as the sum ofall integrated peaks within the retention time window of 0-30.0 min, providing an estimate of the relative abundance of the tentatively identifiedcompounds in the sample.

[2] National Institute of Standards and Technology (NIST) library reverse match score, a mass spectral search score (0–999) that compares an unknownspectrum to a library spectrum. Match factors >900 are excellent, 800-900 are good, and 700-800 are fair.

influenced by chromatogram scaling; when assessed at full scale, all reported peaks remained clearly above the signal-to-noise threshold and were consistently detectable. Collectively, these results indicate that, although oven drying reduces overall signal response, it does not substantially alter the relative distribution or composition of volatile components.

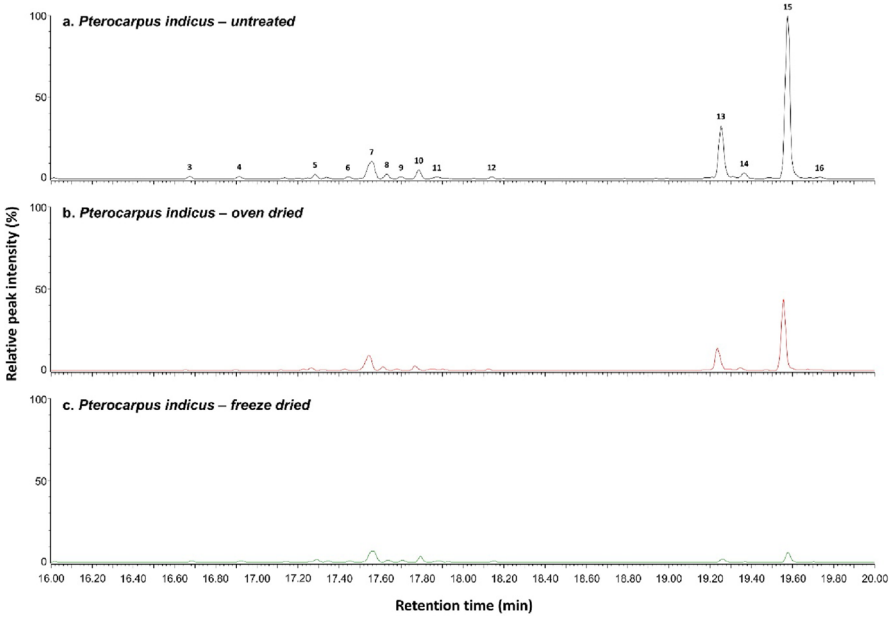


Fig. 2 Total ion chromatograms obtained through HS-SPME GC-MS of a representative sample of *Pterocarpus indicus* following exposure to different treatment conditions: untreated sample (2**a**, black), oven-dried sample (2**b**, red) and freeze-dried sample (2**c**, green). Responses have been normalised to an equivalent scale relative to the largest peak (2**a**, peak 15), with the baseline set to 0%. The retention time window has been narrowed to 16–20 min to allow clearer visual comparison of the principal eluting compounds.

Identification and discrimination of wood species in mixed samples

To assess the suitability of HS-SPME GC-MS as a method of wood identification through headspace sampling, *P. indicus* was mixed with *P. radiata*. This approach aimed to replicate, at a small scale, real-world situations in which traffickers may conceal illicit wood within consignments of legal timber, using pine as a proxy due to its prevalence in trade and known release of odorous volatile organic compounds (VOC). VOC attributable to both species were consistently detected across all mixture ratios tested (0.5:0.5, 0.25:0.75 and 0.1:0.9). The relative emission of compounds associated with *P. indicus* proportionally decreased as the volume of *P. radiata* increased, however both volatilomes were clearly defined even at the highest ratio explored (0.1:0.9, Fig. 3).

Discussion

The potential applicability of HS-SPME GC-MS to complement illicit trade investigations was explored using the tentative characterisation of *P. indicus* as a case study. The analysis revealed a diverse array of VOC, including those known to be associated with rosewood heartwood (Dahat et al. 2021; Krainovic et al. 2018; Zhang et al.

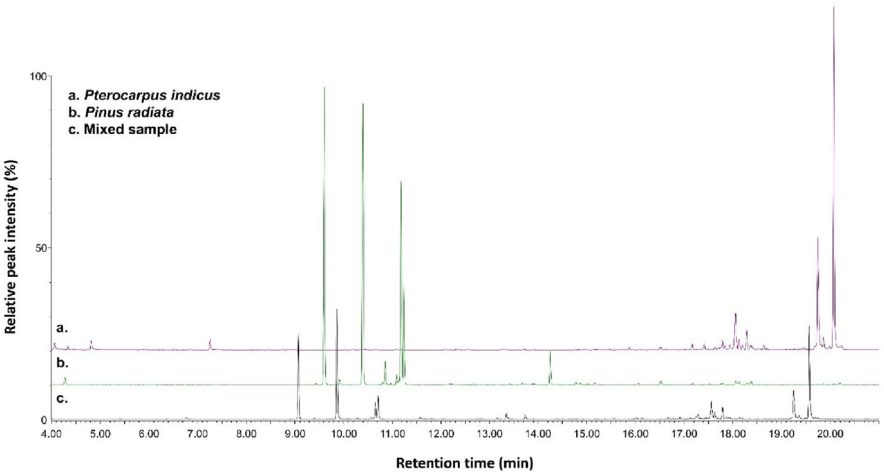


Fig. 3 Total ion chromatograms obtained through HS-SPME GC-MS of a mixed sample (3**c**, black) consisting of powdered *Pterocarpus indicus* (10%) and *Pinus radiata* (90%), with overlaid chromatograms from *P. indicus* (3**a**, purple) and *P. radiata* (3**b**, green) for comparison. The chromatograms are displayed with a 10% overlay step, with each individual trace (3a and 3b) offset by 0.5 min to facilitate visual comparison while preserving peak alignment

2019). GC-MS offers distinct advantages over traditional wood identification methods, such as morphological analysis, DNA-based approaches and DART-TOFMS (UNODC 2016), as it allows for the characterisation of volatile compounds via headspace analysis. VOC profiling could therefore represent a valuable tool in contexts where morphological identification is compromised, such as in processed products, or alternatively where physical samples cannot be readily accessed, such as in our container concealment scenario, offering a versatile method for trace detection purposes. DART-TOFMS, for example, has emerged as a highly effective method for identifying wood, including *Pterocarpus* spp., once a physical sample is obtained (Isak et al. 2025; Lancaster and Espinoza 2012; Price et al. 2021), however it is limited by the need to access the material directly. HS-SPME GC-MS enables non-invasive sampling which could be scaled to analyse air from containers or other restricted environments, potentially facilitating the detection of illicit timber without the need to open or disturb cargo. This approach could also allow for the evaluation of whether target species can be distinguished from legal or commonly co-occurring timber species often used for concealment or misdeclaration in trafficking cases (UNODC 2024; Zavagli 2021).

The predominant compounds identified in this study are similar to those reported in other rosewood species (Dahat et al. 2021; Jiang et al. 2020; Krainovic et al. 2018; Sampaio et al. 2012; Zhang et al. 2019). In particular, the *P. indicus* volatilomes were dominated by oxygenated sesquiterpenes (molecular formula $C_{15}H_{26}O$, RT ~ 19 min), suggesting that these compounds could be important markers for identification purposes. Terpenoids, including sesquiterpenes, are a large and diverse class of plant secondary metabolites that serve a wide range of physiological and ecological functions (Câmara et al. 2024; Wang et al. 2023). While Zhang et al. (2019, 2022a, b)

suggest that spathulenol and pterostilbene are potentially important chemical markers for *Pterocarpus* species identification however, neither of these compounds were identified. Although spathulenol (molecular formula $C_{15}H_{24}O$), an aromadendrane-type sesquiterpene, was not tentatively identified, its presence cannot be ruled out as we observed a strong representation of structurally similar compounds dominating the volatilome profile. Native standards would be required to accurately correlate the identified components of interest - the use of an internal standard or more rigorous quantification approaches would be appropriate for studies seeking to characterise or quantify individual compounds, but these were not required for the exploratory comparisons presented here. In contrast, pterostilbene (molecular formula $C_{16}H_{16}O_3$) was not detected, which is consistent with its limited volatility and poor suitability for headspace analysis by HS-SPME GC-MS, highlighting the differences between solvent-extracted and headspace-derived profiles of untreated wood. Future efforts should aim to expand the HS-SPME GC-MS VOC library relevant to species associated with environmental crime investigations to support detection efforts, particularly as headspace profiles may differ from extracts. Other notable compounds, such as linalool ($C_{10}H_{18}O$), which have been reported in other rosewood species and rosewood essential oil (Krainovic et al. 2018; Sampaio et al. 2012) were not detected in our samples. This may reflect differences in matrix effects and compound release kinetics, however exploring the influence of these factors was beyond the scope of the present study. The objective of this study was not to comprehensively characterise individual chemical constituents, but rather to evaluate whether reproducible and distinguishable volatile profile patterns could be detected for screening purposes. Accordingly, definitive structural confirmation of each component was not critical to the aims of the investigation.

Wood samples were also prepared using two preservation methods, oven (kiln) drying and freeze drying, prior to headspace analysis to assess if these 'traditional' and 'novel, modern' techniques for preparing woods (Elustondo et al. 2023) meaningfully influenced VOC emission. Comparison of the resulting chemical profiles indicated that oven-dried samples most closely resembled untreated material, with only partial reduction of later-eluting compounds identified as oxygenated sesquiterpenes (50%), however, minimal alteration of the spatial distribution of the VOC was observed as a result of this process. Freeze-dried samples consistently exhibited significantly reduced VOC signals, reflecting an apparent decrease in detectable compound concentrations, though again the spatial distribution was consistent. Freeze-drying of wood generally releases fewer VOC compared to high-temperature drying methods as it removes water through sublimation at low temperature and pressure, which may trap or immobilise VOC within the wood matrix (Elustondo et al. 2023; Spadafora et al. 2024). These findings have practical implications for investigations of illicit trade, as wood treatment and preservation methods used could influence the sensitivity of chemical profiling and either facilitate, or hinder, the identification of timber species through headspace analysis. While the use of such preservation methods by traffickers is improbable, where they may more commonly use high-temperature heating to improve stability and defects (Zhang et al. 2022a, b), understanding their effects is valuable when handling seized samples to determine the most suitable method of analytical preparation.

Environmental exposure represents another important consideration for the practical application of this method. In particular, prolonged exposure to elevated temperatures (e.g., containers left in direct sunlight during transport or storage) may influence volatilome profiles. Temperature and humidity within enclosed cargo containers are dynamic and can vary considerably from external environmental conditions (Moloney et al. 2026b). The results of this study indicate that thermal treatment (oven drying) primarily reduced overall signal intensity, particularly among later-eluting compounds, while the relative composition of the profile remained broadly consistent. This suggests a degree of resilience in the compositional signature under moderate thermal stress. However, the effects of extended exposure duration and higher temperatures were not systematically assessed and may result in greater loss of volatile compounds, selective degradation of more labile constituents, or progressive distortion of the profile. Alternatively, despite reduced emission, these compounds may accumulate over time in enclosed environments, thereby sustaining detectable concentrations. Further work is required to evaluate the stability of volatilome signatures under controlled time-temperature conditions, in order to define the thresholds at which analytical performance may be compromised and to support reliable deployment in real-world operational environments.

Building on the profiling of *P. indicus*, the subsequent analysis focused on whether volatiles from this species could still be reliably detected when mixed with a common commercially available wood, pine (*P. radiata*). It is well documented that traffickers frequently conceal high-value illicit hardwoods within consignments of legally traded timber (UNODC 2024; Zavagli 2021), making it important to assess whether HS-SPME GC-MS could reliably identify target volatiles in such mixed scenarios, using pine as a proxy. Three mixture ratios were tested, including as little as 10% *P. indicus* by mass, and in all cases the VOC profiles of both species could still be visually characterised and distinguished in the chromatograms. It should be noted that *P. indicus* and *P. radiata* have relatively distinct volatilomes, wherein the volatiles from hardwoods (i.e. rosewood) and softwoods (i.e. pine) can vary considerably (Taiti et al. 2017) and thus differentiating more closely related species may present a greater challenge. Nevertheless, GC-MS extractive analysis and multivariate analyses has previously proven effective in differentiating *Pterocarpus* spp. and *Dalbergia* spp. (Yin et al. 2018; Zhang et al. 2019), an important consideration where conservation status and legislative protections differ between species. While species-level comparisons were beyond the scope of this study, identification at the family level may provide sufficient evidence to justify a full investigation.

These preliminary findings suggest that volatilome profiling could serve as a complementary tool for identifying *P. indicus* in illegal timber trade monitoring scenarios. The subsequent recommended stage of methods development following these initial small scale characterisation trials should focus on the application of VOC trapping onto sorbent tubes with analysis by Automated Thermal Desorption (ATD) GC-MS. While SPME is excellent in the laboratory scale characterisation, sorbent tube trapping and ATD-GC-MS is a scalable methodology that can be utilised in laboratory and actual container trials without extensive sample preparation. Future work should also aim to expand reference datasets to include samples representing different geographical origins, anatomical regions of wood and ages, to assess how such factors

influence volatilome composition. Biological and environmental factors should be considered when interpreting volatilome data for authentication or forensic purposes, including potential sample contamination from extraneous VOC which can be minimised through careful sampling protocols and post-processing. Emphasis should be placed on headspace air sampling rather than solvent extraction, as the VOC profiles likely differ between these analytical approaches (Yin et al. 2018; Zhang et al. 2019), and realistic profiles which may be obtained from container headspaces are essential. Comparative analyses with related *Pterocarpus* species could further enhance the specificity of chemical markers and support the development of robust forensic or quality control tools. Should this approach progress toward operational trace detection of illegally shipped wood, further targeted characterisation, such as retention index determination, use of reference standards and integration of complementary technologies (e.g. container trials incorporating air-sampling systems such as electronic noses), would form part of a subsequent validation phase. When scaled, GC-MS analysis of the headspace extracted from cargo containers could provide a non-destructive, rapid tool for detecting and identifying high-value illicit timber to support customs and trade enforcement by flagging shipments that warrant further investigation. Air sampling could provide an initial indication of potential irregularities, which would subsequently be confirmed through complementary analytical methods for species verification, supporting enforcement actions and trade tracing. Ongoing efforts should focus on refining and optimising sampling and analytical methods to reflect realistic operational conditions and assess their applicability to portable air sampling technologies that could be scaled to support customs enforcement efforts. Continued innovation is crucial to disrupt trafficking networks and strengthen efforts to combat environmental crime.

Conclusion

An exploratory HS-SPME GC-MS approach was evaluated for differentiating wood products, suggesting potential application in wood identification for trade investigations especially for sampling materials that are otherwise difficult to access, such as containerised shipments. The initial conceptual proof of the application of air sampling methodology was explored using known samples of *P. indicus*, a species vulnerable to commercial trade pressures, and *P. radiata*, a common legally exported wood, with both displaying visually distinctive chromatographic profiles. Sixteen compounds were identified and tentatively characterised in *P. indicus*, predominately consisting of oxygenated sesquiterpenes and sesquiterpene hydrocarbons, with oven- and freeze-drying resulting in the reduced emission of later-eluting volatiles, suggesting that volatile release is influenced by both treatment and environmental conditions. The ability to discriminate between the two profiles even in a mixed sample suggests that HS-SPME GC-MS may serve as a useful method for exploring the preliminary discriminatory potential of wood identification in air samples. When collected from enclosed spaces, this information could provide an initial indication of potential trafficking activity when observed chemical profiles deviate from those expected for declared cargo. Further validation is needed to assess the feasibility of scaling this

approach from small-scale containers to full-sized shipments. Such studies should also evaluate the effects of sample size, extraction methods, the presence of other common wood types and concealment strategies on classification outcomes.

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Data availability All data supporting the findings of this study are available within the paper, with additional data available from the corresponding author upon reasonable request.

Declarations

Conflict of interest The authors declare no competing interests.

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