

## A Review Study of Agarwood Oil and Its Quality Analysis

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### Graphical abstract



### Abstract

This paper presents an overview of analysis agarwood oil and its quality grading. The review suggested agarwood oil can be graded according to their chemical properties and so that there is a common standard recognized worldwide on grading the agarwood oil. Analysis based on chemical profiles is required to ensure that agarwood oil can be classified based on their respective classes or grades where the accurate results can be measured. Conventionally, the grading of agarwood oil is performed by trained human graders (sensory panels) depends on its physical appearance such as color, odor, high fixative and consumer perception. However, this method is limited due to human nose cannot accept many samples in one time and easily get fatigues especially when dealing with continuous production. The human sensory panel also limited in terms of subjectivity, poor reproducibility, time consumption and large labour expense. These are constraining factors in increasing agarwood oil trade and market penetration.

**Keywords:** Grading; agarwood oil; chemical compounds; signal processing

### Abstrak

Kertas ini membentangkan gambaran keseluruhan mengenai analisis minyak gaharu dan pengredan kualitinya. Kajian mencadangkan minyak gaharu boleh digredkan mengikut sifat-sifat kimia dan juga dicadangkan supaya diadakan satu standard yang diiktiraf di seluruh dunia untuk pengredan minyak gaharu. Analisis berdasarkan profil bahan kimia diperlukan untuk memastikan agar minyak gaharu boleh diklasifikasikan berdasarkan kelas atau gred supaya keputusan yang tepat boleh dicapai. Konvensional, pengredan minyak gaharu dilakukan oleh pengred manusia terlatih (panel deria) iaitu bergantung kepada rupa fizikal minyak tersebut seperti warna, bau, persepsi dan gambaran yang tinggi oleh pengguna. Walau bagaimanapun, kaedah ini adalah terhad disebabkan oleh hidung manusia tidak boleh menerima banyak sampel dalam satu masa dan dengan cepat letih terutama apabila berurusan dengan pengeluaran berterusan. Panel deria manusia juga terhad dari segi subjektiviti, keboleholangan yang terhad, penggunaan masa dan perbelanjaan buruh yang besar. Ini adalah faktor-faktor yang mengekang dalam meningkatkan perdagangan minyak gaharu dan penembusan pasaran.

**Kata kunci:** Gred; minyak gaharu; bahan kimia; pemprosesan isyarat

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### 1.0 INTRODUCTION

Agarwood oil is the oil extracted from agarwood trees. Agarwood or *gaharu* is the resin impregnated heartwood of the *Aquilaria* species, a genus which belongs taxonomically to the *Thymelaeaceae* family. The agarwood oil is highly demanded due to its special usage; an incense for religious ceremony, in perfume and traditional medicine preparations [1]. In the Middle East, it is a symbol of wealth and widely used during the wedding ceremony [2-7].

The agarwood oil is traded according to its quality. High quality oil is expensive and low quality is cheap. The oil class also traded based on its physical properties; color and odor. Usually,

dark color and long lasting odor are classified as high quality and sold at premier price [8]. It is normal for a high quality oils to cost between USD126 to USD633 per tola (12 ml) [2]. The wood prices for low qualities are USD19 per kg and up to USD100,000 per kg for superior quality [1, 9].

Many studies have been carried out to analyze the quality of agarwood oil [10-14]. Researchers from Japan has classified Kanankoh as the highest quality among many types of agarwood oil and Jinkoh as the low quality [11, 13, 14]. The relationship between high and low quality of oil has been widely investigated by comparing their chemical compositions [13, 14]. One of the investigations found that the abundances (percentage relative peak

area measured by GC-MS) of the same compound in high quality oil is more than in low quality [14].

There are many techniques available to analyse the chemical compounds of essential oils; electronic nose (Enose), gas chromatography (GC), gas chromatography/mass spectrometric (GC/MS), solid phase micro extraction (SPME), gas chromatography –flame ionization detector (GC-FID), gas chromatography-olfactometry (GC-O) and comprehensive two dimensional gas chromatography (GCxGC) [12, 14-19]. GC-O is the odor-compounds extraction method with combining gas chromatography and human sensory panel [19]. This method is limited to the subject of fairness since human nose cannot tolerate with many samples at the same time [15]. In GC x GC, chemical compounds are separated by a single column based on its properties; the size, length and stationary phase. In GC-FID, the flame ionization detector was used to sense any molecule with a carbon-hydrogen bond. However, it provides poor response to compounds; H<sub>2</sub>S, CCl<sub>4</sub> or NH<sub>3</sub>. The technique is mass sensitive but not concentration sensitive. Furthermore, the flame ionization detectors require a high data rate at 200 Hz to operate [15]. The GC/MS technique is proven and has shown its promising result in analysing the chemical compounds of agarwood oil [8, 10, 13, 14, 16-18, 20-23]. The GC/MS technique combines gas chromatograph and mass spectrometry. The compounds mixture was separated by gas chromatograph and mass spectroscopy was used to characterize every component individually [15]. The other popular method is SPME where it was preferred to detect the odor compounds of agarwood oil [19, 24]. The SPME has produced reliable analysis in many studies since it can give fast result, sensitive, inexpensive, easy to handle, solvent free and selective [19, 24-31].

Based on practice and literature, it was found that there are two ways in grading the agarwood oil. Some countries use term quality of agarwood oil; high and low and some countries use term grade of agarwood oil; A, B, C and D. Each term is applied based on agarwood oil physical properties like color, odor and consumer perception [1, 7, 8, 13].

Technically, the grading of agarwood oil is performed by trained human graders (sensory panels) depends on its physical appearance; color, odor, high fixative and consumer perception [5, 6, 32-35]. However, this method is limited due to human nose cannot accept many samples in one time and easily get fatigues especially when dealing with continuous production [6]. The human sensory panel also limited in terms of subjectivity, poor reproducibility, time consumption and large labour expense [8]. These are constraining factors in increasing agarwood oil trade and market penetration [9].

A review from literature domain suggested agarwood oil can be graded according to their chemical properties and so that there is a common standard recognized worldwide on grading the agarwood oil [9]. Analysis based on chemical profiles is required to ensure that agarwood oil can be classified based on their respective classes or grades where the accurate results can be measured [9, 11, 13, 36].

## 2.0 AGARWOOD OIL

*Aquilaria* is the resinous part of wood and it is known as agarwood, eaglewood, oudh, oud, kanankoh, kyara, jinkoh and kalambak. Taxonomically, the wood comes from *Thymelaeaceae* family. It had been reported that there are nineteen species of *Aquilaria* found in the Asean region [1]. The agarwood has multiple usage from trunk, branches, chips, flakes of uniform quality powder to essential oil [16].

Many studies have shown that there has been a dramatic increase in agarwood oil usage in Malaysia [2, 3, 5, 6, 15, 20, 37-40]. Correspondingly, there is an increasing number of agarwood plantation in this country and the market demand of its oil from all over the world [34, 35]. Agarwood oil is concentrated volatile aromatic compounds produced by agarwood plant, extracted from the stem. Many researchers have highlighted that agarwood oil is produced as incense, perfumery and for traditional medicines [1, 16, 19, 41]. The agarwood oil is traded worldwide and getting high market demand especially from United Arab Emirates, Saudi Arabia, China and Japan. The agarwood oils are traded differently depending on its grade.

The demand of agarwood oil is high in the Middle East, where the oil symbolizes the society ranking, wealthiness and hospitality. It is an important ingredient in the perfumery industry and to generate aroma in wedding ceremonies or banquets [1, 2, 22, 35, 39, 42-44].

### 2.1 The Chemical Compounds of Agarwood Oils

Many researchers had studied the differences of chemical composition in agarwood oil [2, 13, 14, 16, 18, 19, 22, 23, 38, 40, 45]. Most of them agreed that sesquiterpenes components and its chromone derivative were the major compounds in agarwood oil [2, 13, 14, 16, 19, 23, 40]. The composition of the compounds, determine the high and low quality of agarwood oil [13, 14]. Forest Research Institute Malaysia (FRIM) has carried out classification of agarwood oil quality based on wood physical properties, long lasting aroma when burnt, color, resin content, high fixative properties and consumer perception [2]. For example basis odor and appearance can be applied to grade different quality of the agarwood oil falling from A to D [2, 23]. A research in Japan meanwhile has classified the quality of agarwood oil either high or low quality based on peak area percentage (or abundances) of chemical compounds. The wood oil which contain  $\alpha$ -guaiene with the peak area less than 0.05% classified as a high quality oil however the wood oil which is not containing  $\alpha$ -guaiene classified as low quality [13].

The highest quality of agarwood oil has a lot of resin which means that it contains various kind of oxygenated sesquiterpenes and chromone derivatives [11]. Three compounds of sesquiterpenes are detected only in the highest quality. They are (-)-*guaia-1(10),11-dien-15-al*, (-)-*selina-3,11-dien,9-one* and (+)-*selina-3,11-dien,9-ol* [12]. Reflective to that, the presence of sesquiterpenes differentiate the premium of agarwood oil in the market [12]. The grade A (also called as supreme or deluxe) agarwood oil is trading expensively at a cost between RM400 to RM2000 per tola (12 ml) [2, 15, 37].

### 2.2 The Agarwood Oil Quality Analysis

There are different techniques being used to analyse the quality of agarwood oil (i.e. high and low); gas chromatograph (GC) charts [13, 14], qualitative and quantitative difference (or chemical composition) table [19] and thin layer chromatogram (TLC) [22]. By using GC chart, the differences of relative peak area (%) for sesquiterpenes components in high and low quality oil can be observed visually [13, 14]. Various guaiene and eudesmane sesquiterpenes were found in high quality however most of them were not detected in low quality of agarwood oil [14]. Benzaldehyde and anisaldehyde compounds in high quality (i.e. Kanankoh) were found less than in low quality (i.e. Jinkoh) [13]. The components; 9-11-eremophiladien-8-one and oxo-agarospirol were found in low quality agarwood oil.  $\beta$ -agarofuran,  $\alpha$ -agarofuran and 10-epi- $\gamma$ -eudesmol were the major compounds in high quality [2, 14, 19, 40]. Among them,  $\beta$ -agarofuran is the

most important compound in high quality and it is contributed to the aroma of agarwood oil [19]. Some monoterpenes were not exist in high quality oil [13] which contain high resin content [22]. The empirical study showed that the characteristic spots with deeper color in TLC figures can be related to the high quality of agarwood oil [11]. Table 1 summarizes the component based characteristic of agarwood oil.

**Table 1** Component based characteristic of agarwood oil

Component	Low Quality	High Quality	Ref
1. resin content	low	high	[23]
2. $\alpha$ -guaiene	no	yes	[18]
3. (-)-guaia-1(10),11-dien-15-al, (-)-selina-3,11-dien,9-one and (+)-selina-3,11-dien,9-ol	no	yes	[24]
4. guaie and eudesmane	no	yes	[22]
5. benzaldehyde and anisaldehyde	less amount	more amount	[18]
6. 9-11-eremophiladien-8-one and oxo-agarospirol	yes	no	[7]
7. $\beta$ -agarofuran, $\alpha$ -agarofuran and 10-epi- $\gamma$ -eudesmol	not mentioned	yes	[17]
8. $\beta$ -agarofuran	not mentioned	the most important compound	[19]
9. sesquiterpenes	not mentioned	major	[1, 13, 19, 22]

### 2.3 The Agarwood Oil Grading Method

Conventionally, the agarwood oil was graded according to its colour, aroma and fixative based on human's sense [2, 40]. However, it was difficult to standardise it's quality from the aroma due to human nose cannot smell many samples continuously [2, 3, 17, 19, 38, 44-46]. There were also limitation in terms of subjectivity, poor reproducibility, time consumption and expensive labor cost [47].

There are various grading methods applicable for agarwood oil. Different country has its own way to grade/classify the agarwood oil. Agarwood oil is traded on the basis of qualities; high and low but there are some countries prefer grades. In Malaysia, agarwood oil is classified according to the grade of A, B, C and D [7, 8]. In India, they use grades too; A, B, C and D but were based on the color of agarwood oil [1]. In Japan, agarwood oil is categorized as high and low quality where Kanankoh is the highest quality and Jinkoh is low quality [13]. Therefore, no standard available for this highly price oil [15].

Laboratory technique; GC-MS analysis was carried out to grade agarwood oil by analysing the chemical profiles of the oil [11-13, 19, 45]. GC-MS analysis identifies the significant existence of common compositions; valerianol [2, 23], pentadecanoic, tetradecanoic, dodecanoic and hexadecanoic acid [23], elemol [2, 38, 40],  $\beta$ -agarofuran,  $\alpha$ -agarofuran, epi- $\gamma$ -eudesmol and agarospirol and  $\alpha$ -guaiene [2, 12]. The analysis characterized the volatile compounds of agarwood oil to different quality and showing that there were differences of oil

compositions in agarwood oil [12, 13, 19, 36, 38, 48]. For example, agarospirol and  $\beta$ -agarofuran as Type A and  $\alpha$ -agarofuran, 10-epi- $\gamma$ -eudesmol and oxo-agarospirol as Type B [36].

Another grading method is by analysing the odour of the agarwood oil. The GC-MS helps to relate the compounds and odour based on odoriferous properties in agarwood oil [2, 12, 19]. For example, (-)-guaia-1(10), 11-dien-15-al has a pleasant note,  $\beta$ -damascenone like woody note with a touch of camphor and (-)-selina-3, 11-dien-9-one has a fresh and sweet odour to represent gorgeous and elegant character of agarwood oil [12].

Instead of odour, colour has been used to grade the agarwood oil; different colour corresponds to different quality of oil [11-13]. Dark colour (especially black) refer to the high quality with very expensive market price [40]. Researches on colour in oil volatile components have been carried out and they found that different colour has different chemical constituents [38, 45]. For an example, GC-MS found that oxidoagarochromone as a pale yellow in oil component [49].

Both colour and odour has a complex mixture of sesquiterpenes and chromone derivatives [15]. As major volatile components, the presence of sesquiterpenes is popular in market [16].

### 3.0 AGARWOOD OIL ANALYSIS TECHNIQUE

There are many techniques applied to analyze the chemical composition in essential oil [2, 13, 14, 16, 18, 19, 22, 23, 38, 40, 45]. Some of them are electronic nose (Enose) [3, 31, 50, 51], gas chromatography (GC), gas chromatography-mass spectrometric (GC-MS), solid phase micro extraction (SPME), gas chromatography-flame ionization detector (GC-FID), gas chromatography-olfactometry (GC-O) [12, 14, 16-19] and comprehensive two dimensional gas chromatography (GC x GC) [15, 39].

Electronic nose or Enose has been applied in wide range of applications; in food, beverages and cosmetics industries [3, 28, 52-59]. It is used to classify the quality of essential oil and food [3, 52, 53, 60, 61], to discriminate the different blends, to pattern recognition the aroma [50, 60] and to determine the odour discrimination in cosmetics [1] and agarwood oil [3, 28, 52, 56-59, 62]. Besides that, Enose is able to monitor the modified atmosphere (MA) for different temperature regimes especially in food [8].

GC-O is the odor-compounds extraction method which combine gas chromatography and human sensory panel [19, 63]. This hybrid method was applied to measure the sensory activity of chemical, to process human olfactory and to detect the chemical compounds in fruit [63, 64]. The GC-O is depends on human nose which can influence the description on the odor quality of the oil [64] Thus, it is limited to the subject of fairness since human nose cannot tolerate with too many samples at the same time [15].

The other analysis method is GC x GC. The first concept of this method was introduced by Giddings in 1984 before it is being used in petroleum, food and fragrance analysis [39]. By using this technique chemical compounds are separated by a single column based on its properties; the size, length and stationary phase. The detector for this analysis should has a high rate of data acquisition so that the system can respond very fast enough during the evaluation [15]. The concept in GC-FID is the flame ionization detector applicable to sense any molecule with a carbon-hydrogen bond [15]. This analysis was performed to extract the chemical compositions in essential oils, fatty acids and chemical products [38]. However, it provides poor response or not all to compounds; H<sub>2</sub>S, CCl<sub>4</sub> or NH<sub>3</sub>. The technique is mass sensitive but not

concentration sensitive. Furthermore, the flame ionization detectors require a high data rate at 200 Hz to operate [15].

The GC-MS technique is proven and has shown its promising result in analysing the chemical compounds of agarwood oil [8, 10, 13, 14, 16-18, 20-23]. The concept of GC/MS is combining two techniques; gas chromatograph and mass spectrometry. The compounds mixture was separated by gas chromatograph and mass spectroscopy was used to characterize every component individually [15].

The other preferred method is SPME detecting the odour compounds of agarwood oil [19, 24]. The SPME has shown as a reliable analysis in many studies since it can give fast result, sensitive, inexpensive, easy to handle, solvent free and selective.

### 3.1 The GC-MS Analysis

The GC/MS is a well known, easy, fast and proven method to study the chemical profiles in agarwood oil [2, 38, 45]. This technique has shown its capability by discriminating the chemical compounds from the herbal material [65] and identify the most abundances in fruits [64].

The GC/MS has been used to successfully determine the oil composition from agarwood smoke by heating [13]. The analyses of GC and GC/MS showed the existence of major sesquiterpenes and its chromone derivatives in agarwood oil [38]. Different compositions marked in the agarwood oil using GC and GC/MS will establish whether the oils are generated from healthy wood or artificially inoculated wood [23]. Different oil grade applies even though there are similarities in agarwood oil sampled through GC profiles but different in percentage concentration from GC/MS analysis [2].

### 3.2 The SPME Analysis

The aroma compounds of essential oils have caught interest of many researchers in identifying their volatile profiles [9, 15, 19, 24, 25, 29, 52, 66, 67]. One of the famous methods for this study is solid phase microextraction (SPME) which has been proven its effectiveness in various application; plants, food, beverage biological and environmental samples [15, 25, 29, 52]. It is also used to identify the agarwood oil compounds [9, 19, 24]. The chemical compounds identified using this technique are useful to ensure the accuracy in grading agarwood based on their chemical properties [9].

The SPME is recognized as a fast, solvent free, easy to use and sensitive method to isolate the volatile chemical compounds of essential oil [15, 24]. The technique was determined by a fiber existence where different fibers result in different sensitivity [15, 19].

The SPME fiber is reported to be many types; divinylbenzene-carboxen-polydimethylsiloxane (DVB-CAR-PDMS), polydimethylsiloxane-divinylbenzene (PDMS-DVB), polydimethylsiloxane (PDMS) and polyacrylate (PA) [15, 19, 24]. Several studies showed that the oil compounds vary according to different fibers [9, 19, 29]. This is because every fiber has different properties that rely on their thickness and chemical properties [9]. A study has shown that PA fiber has low dispersion and CAR/PDMS has high sensitivity [15]. Other studies found that DVB-CAR-PDMS fiber extracted the highest number of volatile compounds compared to others [15, 19].

The analysis technique of agarwood oil; Enose, GC, GC-MS, SPME GC-FID and GC-O will be producing a lot of chemical compounds from the extraction [19, 23, 38]. In order to do further analysis; signal processing, not all compounds are being used. Data transformation should be applied so that only significant chemical compounds will be utilized; classification and modelling

[68, 69]. The application of data transformation will reduce the sample size of data collection from a large number to a smaller number of data [70]. Besides that, the technique also benefit in term of economical reason [69].

## 4.0 CONCLUSION

From the above review, it can be seen that agarwood oil is an important commodity and its trade highly dependent on its quality and/or grade. Currently the grading of agarwood oil is done manually which is based on its physical appearances; colour and odour. However, there were studies mentioned that chemical compounds of agarwood oil can be used to decide the quality of agarwood oil, since they found that agarwood oil were made up of complex mixture of sesquiterpenes, oxygenated sesquiterpenes and its chromone derivation. Thus, the agarwood oil classification system using these chemical components can be used to provide fast, robust, automatic and accurate result. The review found that GC-MS and SPME techniques provided many advantages; well established, easy to use, fast, sensitive method and solvent free analyzer.

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