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# Changes in rosewood (*Aniba rosaeodora* Ducke) essential oil in response to management of commercial plantations in Central Amazonia

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ABSTRACT

Rosewood essential oil (REO) is an Amazonian industrial crop required by fragrance and cosmetic industries worldwide. This essential oil (EO) is obtained from a singular resource, the endangered tree species *Aniba rosaeodora* Ducke. The management of this resource influences the chemical composition of the EO, affecting the quality and international price of the product. A systematic study was performed within the rosewood plantations of two major REO producers. Chemical composition (GC–MS) and REO yields were analyzed to identify the best harvesting periods and the potential sustainable use of other plant parts, such as resprouting shoots, to produce the oil. With a large sample and a well-controlled statistical approach, the study's methodology allowed us to describe the differences in the REO composition between tree parts and between harvest times. REO yield was highest in branches from the first harvest and in resprouting leaves from the second harvest. In the first harvest, α-pinene was found only in REO from branches and leaves, and cyclosativene was sourced only from branches, regardless of the sampling region. Geraniol was detected only in the first-harvest REOs, while myrcenol was found only in second-harvest REOs. The temporal spacing of harvest rotations and the use of different plant parts in extraction are the main management tools determining the variations in REO. Despite higher EO yield in the stem, the management by crown pruning assures sustainable oil production. Greater understanding of these variations may provide opportunities to expand the production chain of globally exported REO.

## 1. Introduction

Rosewood (*Aniba rosaeodora* Ducke - Lauraceae) is an Amazonian tree threatened with extinction (IBAMA 1992; CITES 2010; IUCN 2014). Since wild populations are under full protection (directive N$^0$ 443 12/2014, MMA), commercial rosewood plantations (formed from genetic material of natural populations) are meeting the global cosmetic industry's demand for the essential oil (EO) derived from this species. These plantations not only regulate the exploitation of rosewood and reduce the pressure on wild populations, but also generate jobs and development in rural areas of the Central Amazon (McEwan et al., 2016). International demand for Brazilian REO has been constant, with untapped growth potential in the world market. Consequently, many producers are interested in commercial rosewood production; however, they encounter a lack of technical information regarding production management (May and Barata, 2004) and a lack of clarity in the standardization established by the current legislation.

Although the development of technical criteria for rosewood management is recommended by law (IN N$^0$ 02/2006, SDS), there is a regrettable lack of studies on the effectiveness of harvesting patterns to optimize REO extraction under commercial production conditions, and on the influence of particular harvesting patterns on the yield and quality of the final product.

Above-ground biomass management (ABM) is a harvesting technique whereby trees are either coppiced (cut at 50 cm above the soil) or pollarded (100% of their crowns pruned) and are then allowed to regrow (Sampaio et al., 2005). Managing above-ground biomass does not require intensive preparatory operations (as is the case with seedling planting), and the care requirements are simple and provide quick benefits (Spinelli et al., 2017). Given the difficulties in obtaining rosewood seedlings, regrowth is the best management option for commercial plantations. However, one consequence of this management practice is that the regrowth differs in terms of nutrient status (Krainovic et al., 2017a). Studies on other species have observed that

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ABM causes differences in physiological mechanisms (Moreira et al., 2012; Shibata et al., 2016; Pausas et al., 2004, 2016). For rosewood, ABM could potentially result in differences in secondary metabolite production, notably terpenes, causing variation in the final product required by industries, though the extent and nature of such changes have not been previously studied. Sequential management (harvest and resprouting) of commercial plantations requires that the characteristics of the regrowth meet the needs of the market. Currently, a diverse chemical composition may diminish the quality of the REO to below the industrial standard for its fixative and perfuming activities, thereby reducing its commercial value. Nevertheless, it could also create new opportunities (new products).

The production chain of REO requires a full and integrated understanding of plantation management, from proper tree management to the production of quality REO for the international market. The extent of compositional variability of REO from managed plantations, as well as the factors that can influence this variability, is key information. Although REO consists mostly of low molecular weight monoterpenes and sesquiterpenes—the major constituent is linalool (78–93%) (Chantraine et al., 2009; Krainovic, 2011; Fidelis et al., 2012, 2013)—a range of minor components confer the precise fragrance bouquet unique to REO. Consequently, the conditions under which a rosewood plant is grown must be able to stimulate the redirection of metabolic pathways, resulting in the biosynthesis of different compounds (Morais, 2009). The present study is the first to provide relevant information on the compositional differentiation of REOs cultivated in Central Amazonia and the influence of above-ground biomass management on their chemical composition. The study is based on the largest sample ever used in an analysis of rosewood essential oil production.

The study hypotheses were that (1) the cultivation region influences the yield and chemical composition of REO; (2) the use of different tree parts for REO extraction will produce different yields and chemical compositions of the final product; and (3) the use of regenerated biomass will yield REOs with different characteristics than REOs extracted from biomass of the first harvest in a sequential management system. Consequently, the objectives of the current study were to (1) understand the effects of the cultivation region on REO composition and yield; (2) characterize the variation in REOs from different parts of rosewood trees; and (3) investigate the feasibility of sequential harvesting of commercial rosewood, based on yield and chemical composition of the REO.

## 2. Methods

### 2.1. Site descriptions and plant materials

The study was conducted in three rural areas. Two of these areas (homogeneous plantations, 10 and 12 years old, hereafter referred to as C 10 and C 12, respectively) are located in the municipality of Maués (350 km by river from Manaus), and the third area (17-year-old plantation in lines traversing the natural vegetation, hereafter C 17) is in the municipality of Novo Aripuanã (469 km by river from Manaus). Both municipalities are in the state of Amazonas, in Central Amazonia (Fittkau et al., 1975), Brazil (Fig. 1). The climate in Maués is hot and humid, with regular and abundant rainfall, an annual rainfall of 2,101 mm, and an annual mean temperature of 27.2 °C. According to Köppen-Geiger, the climate is type Amazonia Af. The soil under rosewood plantations is classified as dystrophic yellow red latosol (Krainovic, 2011). The climate of Novo Aripuanã is also classified by Köppen-Geiger as type Af, hot and humid, with an annual average rainfall of 2,444 mm and an annual mean temperature of 26.9 °C (Kottek et al., 2006; http://en.climate-data.org/). Predominant soils in the region are classified as yellow poor oxisols saturated with oxidized iron and aluminum with low pH (Tanaka and Vieira, 2006).

### 2.2. Field sampling, harvesting and extraction of essential oils

In each area, samples of plant material (stem, branches and leaves) were collected from 48 trees by cutting the stems of 36 trees at 50 cm above soil level (coppicing), and by pruning 100% of the crown (pollarding) of the other 12 trees. Sampling occurred on two occasions: i) first harvest and ii) second harvest. Stems were then sectioned into discs for the collection of wood samples. Branches and leaves were sampled at the four cardinal points, at the height of the middle third of the tree crown. Sampling this way homogenized the collection in relation to incident sunlight, since the crowns of C 10 and C 12 trees were already touching, while rosewood canopies in C 17 were competing with naturally regenerating vegetation for light. Material was divided into "stem wood", "leaves", "branches", and "branches + leaves" at a 1:1 wt ratio. Material from six trees adjacent to each sampled tree was combined to provide a composite sample for REO extraction. Exactly 12 months after management action, in the same season in each area, a new sampling (second harvest) of all individuals was performed using the same methods, collecting branches and leaves of the resprouting crowns (pollarded trees) and stumps (coppiced trees). We collected a total of 90 samples in the first harvest and 72 samples in the second harvest.

Collected samples were stored in paper bags to avoid exposure of the material to light and the consequent loss of volatile constituents. After pre-drying in the shade and at room temperature for 72 h (a duration traditionally used by rosewood oil producers), the samples were ground and then stored in plastic bags in a freezer at −15 °C. To minimize any possible variation, a randomized block was used to establish the extraction sequence. REO was extracted by hydrodistillation using a modified Clevenger apparatus. Samples were weighed for REO yield calculation and then placed with distilled water in a glass flask of known capacity and hydrodistilled for two hours (timed from the first drop of REO at a temperature compatible with the gentle boiling of the material within the flask). All extractions were performed using the same homeothermic blankets and in the shortest time possible (45 days) to reduce variations that could influence distillation results. After each distillation, the Clevenger apparatus was washed with 80% hexane–ethyl acetate solution. Samples of REO + water resulting from hydrodistillation were stored in a freezer at 6 °C for 24 h; they were subsequently dried using anhydrous sodium sulfate ($Na_2SO_4$). The dried REOs were stored at a temperature of 6 °C prior to chromatographic analysis. REO yield ($Y\%$) was calculated based on the ratio between the weight of the sample and the weight of the extracted REO:

$$Y_{\%} = (essential\ oil\ weight/sample\ weight)*100 \quad (1)$$

### 2.3. GC-FID analysis

Analyses were conducted with a flame ionization detector (GC-FID) in a THERMO TRACE 1310 chromatograph, using a capillary column with 100% dimethylpolysiloxane stationary phase (25 m × 0.25 mm, film thickness 0.25 mm) and helium as the carrier gas, at a flow rate of 2 ml/min (model DB-5). The chromatograph was programmed as follows: isothermal at 80 °C for 1 min; 80–150 °C at 12 °C/min; 150–180 °C at a 6 °C/min; isothermal at 180 °C for 1 min; and a total optimal run time of 12.8 min, reducing analysis time of the repetition samples. Samples were injected at a concentration of 5 mg of REO to 1 ml of 80% hexane-ethyl acetate solvent. The injection port was adjusted to 220 °C using a ratio of 1:4, and the detector temperature was set at 240 °C. Given the extreme olfactory and physical characteristics (coloration) of the REOs, chromatographic run optimization was conducted with the aim of minimizing analysis time without loss of chromatogram resolution. Consequently, we tested chromatographic analyses with different rates of temperature variation (°C/min) and ran analyses with and without isotherms (Table A.1). The chosen method was suitable for the essential oils extracted from both the lighter leaf-based material