



Steam distillation of mallee leaf: Extraction of 1,8-cineole and changes in the fuel properties of spent biomass



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HIGHLIGHTS

- In the first 15 min, extraction of 1,8-cineole in leaf via steam follows zero-order kinetics.
- After 15 min, extraction of 1,8-cineole in leaf via steam follows first-order kinetics.
- Steam distillation removes some organic and inorganic matters in leaf.
- Proximate and ultimate analyses of the raw and spent biomass are similar.

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ABSTRACT

Mallee biomass is an important renewable energy source for the future sustainable energy supply in Western Australia. Embedded in mallee leaf, 1,8-cineole is a value-added product that can be extracted for further improving the economic performance of an envisaged mallee-based biomass supply chain. This study reports the steam extraction of 1,8-cineole from mallee leaf for 1–180 min and its effect on the fuel properties of the spent biomass. The experimental results show that in the first 15 min, steam distillation extracts ~90% of 1,8-cineole rapidly and the extraction follows zero-order kinetics, regulated by the rate of latent heat (provided by steam) for 1,8-cineole evaporation. After 15 min, extraction is slow and follows first-order kinetics, controlled by the slow internal diffusion of the residual 1,8-cineole. Overall, 1,8-cineole extraction virtually completed after steam distillation for 60 min. Steam distillation leads to little changes in the proximate and ultimate analyses and hence mass energy density of the spent leaf. However, it removes some total organic carbon and significant amount of Mg, Ca, and Cl in the spent leaf via interactions between the leaf and the steam (and/or reflux). It has also changed the chemical association of Mg and Ca with the chemical structure of organic matter in the spent leaf.

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1. Introduction

Biomass as a renewable energy source plays significant roles in future sustainable energy supply [1,2]. In Western Australia (WA), mallee eucalypts are planted in agricultural land for managing dry-land salinity that seriously threatens the State's sustainable food production [3]. As a biomass, mallee can be produced at low cost, on a large scale and with low energy and carbon footprints [4,5] hence potentially an important feedstock for energy applications (e.g. via various technical routes [6–11]). The leaves of mallees contain eucalyptus essential oil that is widely used in medicinal, perfumery and flavouring applications and 1,8-cineole is the most abundant (up to 96%) constituent in the oil [12–18]. Therefore,

producing 1,8-cineole as a value-added product from mallee leaf is an important strategy to further improve the economic performance of an envisaged mallee-based biomass supply chain in WA [3,19,20].

Extraction of essential oil containing 1,8-cineole from mallee leaf can be commonly done via hydrodistillation or steam distillation, both of which are environmentally friendly, safe to operate and easy to scale up [21–36]. In comparison to hydrodistillation, steam distillation achieves higher oil yield and minimises the extraction of impurities and the loss of polar compounds [37,38]. After either hydrodistillation or steam distillation, the spent leaf following the extraction of 1,8-cineole is potentially an important feedstock in a future mallee-based bioenergy supply chain system [36]. Previous studies reported that the extraction of 1,8-cineole from eucalyptus leaves via steam distillation is slow and requires a long period of steam distillation (typically at a scale of several

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hours) [28,30]. Unfortunately, to the best of our knowledge, no kinetic data on the extraction of 1,8-cineole from mallee leaf via steam distillation has been reported. There are also no data reported on the fuel properties of the spent leaf after steam distillation.

Therefore, this study is focused on the extraction behaviour of 1,8-cineole during steam distillation and the effect of such steam distillation on the fuel properties of spent biomass. A series of experiments have been conducted to investigate the steam distillation of mallee leaf at various extraction time (1–180 min). The extraction kinetics of 1,8-cineole are then derived and the fuel properties of spent leaf samples are also characterised. The effect of steam distillation on the loss of fuel materials and the occurrence of inorganic species in the spent leaf are also studied.

2. Materials and methodology

2.1. Materials

Leaves were collected from mallee trees (five-year old, *Eucalyptus polybractea*) cultivated in the Narrogin region in the wheatbelt area of WA. The leaves were then dried in an oven at 40 °C to reduce the moisture content to ~5% and cut to ~1 cm × ~1 cm (length × width). Hereafter, the leaf sample after drying and cutting is referred to as “raw leaf”.

2.2. Steam distillation experiments

Steam distillations were carried out using a laboratory setup (see Fig. 1) that consists of an extraction column (inner diameter: ~9 cm), a flask, a heating mantle and a condenser. Milli Q water was heated in the flask to continuously produce steam which flowed through the extraction column containing the mallee leaves. In each experiment, ~10 g of the raw leaf sample was charged into the extraction column. In all experiments, the steam flow was maintained at 1.3 kg/min/m², the highest value allowed by the experimental system. A series of extraction experiments were carried out at various distillation time ranging from 1 to 180 min. In all experiments, the extraction column was wrapped with heating tape and insulation in order to maintain the temperature inside the extraction column at 105 °C. Once each distillation was completed, the flask and the column were separated immediately and the leaf

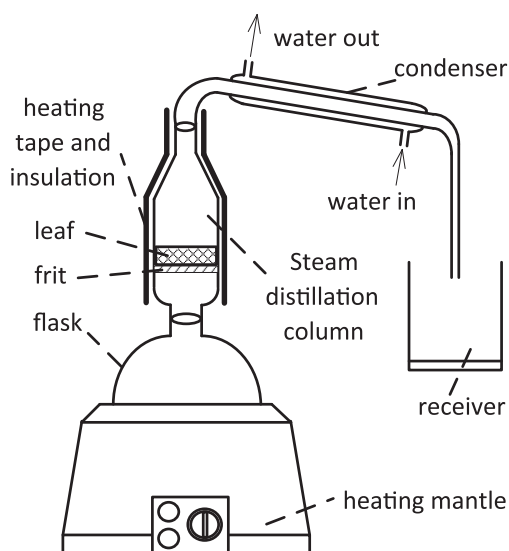


Fig. 1. Experimental setup used for mallee leaf steam distillation.

sample (termed as “spent leaf” hereafter) was then collected. All experiments were done at least in duplicates.

2.3. Semi-continuous leaching experiments

Water leaching of the raw and spent leaf samples was carried out using a semi-continuous leaching apparatus, similar to the one used in a previous study [39]. An HPLC pump (Alltech model 627) was used to deliver a stream (100 mL/min) of ultrapure water through a tubular reactor cell. In each experiment, ~25 mg of the raw or spent leaf sample was loaded into the reactor cell that was then sandwiched by two stainless steel mesh filters. The leachate samples were collected at the exit of the reactor cell at various leaching times for subsequent quantification of total organic carbon (TOC), alkali and alkaline earth metallic (AAEM, mainly Na, K, Mg, and Ca) species, and chlorine (Cl). All experiments were done at least in duplicates.

2.4. Sample analysis and characterisation

Proximate analysis of the raw and spent leaf samples were conducted using a thermogravimetric analyser (TGA, model: METTLER) in accordance with ASTM E870-82 [40]. For ultimate analysis, the contents of C, H and N of the raw or spent leaf samples were determined using a CHNO elemental analyser (model: Perkin Elmer 2400 Series II). The content of Cl was determined via a method recently developed [41]. The content of S was quantified using a previous method [42]. The oxygen (O) content was determined by difference.

The concentrations of 1,8-cineole in the raw and spent leaf samples were determined via a previous method [36] combining solvent extraction and quantification using Gas Chromatography (GC). Approximately 3 g of leaf was boiled under reflux for 90 min in a weighed aliquot of approximately 50 mL absolute ethanol containing 0.25% methyl isobutyl ketone (MIBK) as an internal standard. A Hewlett–Packard 5890 GC fitted with a split injector, a flame-ionization detector and capillary column AT-35 (Grace Davidson Discovery Sciences) of 60 m × 0.25 mm × 0.25 μm was used to determine the concentration of cineole in the extracts using both internal and external standard calibration.

The contents of AAEM species in the raw and spent leaf samples were determined using a method detailed previously [36,43]. About 10 mg of a biomass sample (either the raw or spent leaf samples) was ashed in a muffle furnace according to a designed temperature–time program that avoided ignition during the ashing process. The ash was then digested in a Pt crucible using a mixture of concentrated acids (HF:HNO₃ = 1:1) at 120 °C for 12 h. After the evaporation of the excessive acid, the residue remaining in the crucible was then dissolved in 0.02 M methanesulfonic acid solution. The inorganic species in the solution was then quantified via an ion chromatography (IC, model: Dionex ICS-3000; column: IonPac CS12A; guard column: IonPac CG12A) equipped with suppressed conductivity detection system. The concentrations of AAEM species in the leachate produced from semi-continuous leaching were quantified via the same IC. The concentration of Cl in the leachate was analysed using another IC (model: DIONEX ICS-1100; column: IonPac AS22; guard column: IonPac AG22) whereas the total organic carbon (TOC) in the leachate was quantified using a TOC analyser (model: Shimadzu TOC-V_{CPH}).

Morphological characterisations were carried out using a scanning electron microscope (SEM, model: Zeiss EVO 40XVP), the working distance and voltage of which were 5 mm and 15 kV, respectively. Cross-sections were prepared from the raw and spent leaf samples by setting them in epoxy resin which was then solidified, polished and coated with carbon for SEM characterisation, following a similar procedure used in a previous study [44].

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