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Research Article

Wood Fire Detection by Imitating Beetle Olfaction with Gas Sensors

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Abstract

Volatile organic compound (VOC) based fire risk assessment systems for wood land fires can shorten the time between the outbreak of a fire and the arrival of fire crews. This can prevent the development of crown fires, which are harder to control than ground fires. Metal-oxide semiconductor gas sensors derive of good technical properties for VOC detection. In this study, the VOC emissions of heated lignocellulose biomass (*Eucalyptus globulus*) were analyzed by GC-MS. Eucalyptol and furfural showed the highest emission rates. All of the 20 identified VOCs were tested on the antenna of the fire beetle *Merimna atrata* and eucalyptol and furfural both were among the 13 VOCs that were significantly electrophysiological active on the antenna of the beetle. Three semi-conductor metal oxide gas sensors allowed a monitoring of the temperature dependent VOC pattern. The feasibility of such gas sensors for early fire detection is discussed.

Keywords: Electrophysiology; Eucalyptol; Fireriskassessment; Furfural; Insect olfaction; *Merimna atrata*; Semi-conductor metal oxide gas sensors; VOC; 2-methoxyphenol α -pinene

Introduction

Eucalyptus species are grown worldwide in plantations and in some countries already dominate the flora [1]. While the seed release of several eucalypt species is induced by heat as a result of an adaption to frequent wood land fires, these fires are a serious threat for the people. An effective fire management includes measures like prescribed burnings, evacuation plans for inhabited areas, well trained fire crews, and automatized early fire warning systems. It is important to detect a fire early, because when the crowns of the trees ignite the fire will extend very fast. Factors affecting the crown fire risk are a large organic fuel load (e.g. dry litter, grass, bushes, smaller trees, and dried bark), a long period of hot and dry weather, and a high differentiation in the tree heights. In some regions of the world the increasing average temperature favors woodland fires. An improved fire risk assessment can decrease the time interval between the start of a ground fire and the arrival of fire crews, which is crucial to reduce the risk of a crown fire [2,3].

The detection of smoke is an established way to locate fires. Smoke detectors are sensitive for optical cues, particle size distributions, or gaseous products of the thermal degradation of lignocellulose [3,4]. A smoldering fire in a eucalypt plantation is already a high risk. Therefore, an assessment of the fire risk by sensors is more feasible. When forest biomass heats up, the volatile accessory compounds (VOCs: Volatile Organic Compounds) of the wood and the leaves are emitted. These VOCs comprise mainly tree species specific monoterpenes. If the heat increases further, the bonds of lignocellulose macromolecules (mainly hemicelluloses, cellulose, and lignin) will be oxidized incompletely at low lambda values ($\lambda < 1$, reductive or pyrolytic phase) [6-8]. This will release the gases H₂, CO, and VOCs. Cellulose and hemicelluloses predominantly yield VOCs from the chemical group of furans [7-13] and lignin predominantly yields methoxyphenols [5,13,15-18]. The order of VOCs emitted before the ignition can be used as a cue for a metal-oxide semiconductor gas sensor system [3,13,19,20] to assess an increasing fire risk. Established VOC sensors, e.g. metal-oxide semiconductor gas sensors [19,20], lack of sensitivity towards single VOCs in a complex VOC pattern. This is due to their operation principle: An electrical signal is generated by means of a resistance alteration of the sensor surface. The

alteration is caused by the interaction (ionosorption, reduction and/or oxidation) of a VOC with the band structure of the metal-oxide crystal surface. Therefore, metal-oxide semiconductor sensors are selective for different states of VOC oxygenation rather than for the precise chemical structure of one VOC [20]. Sensor arrays combine different metal-oxide semiconductor sensors to enhance the selectivity by means of a signal pattern analysis [21,22].

The selective detection of VOCs specific for heated eucalypt wood and leaves is necessary to assess an increasing fire risk in eucalypt forests [13]. These VOCs should have three characteristics: 1) they should be emitted before the ignition of eucalypt wood and leaves in order to allow a sensor system to assess the fire risk before ignition; 2) they should occur in prominent quantities to be selectively detectable, because metal-oxide semiconductor sensor systems lack of sensitivity towards single VOCs in a VOC composition; and 3) in eucalypt forests they should be emitted exclusively by heated eucalypt wood and/or leaves to avoid false alarms.

Insects are able to detect VOCs with high accuracy, as they depend on their ecological information. The biochemical mechanism of insect olfaction is based on odorant binding proteins (OBPs) in the sensilla on insect antenna. These proteins bind VOCs selectively. The resulting protein/VOC complex can depolarize neurons by activating ion channels via receptor triggered G-coupled protein cascades. This complex biological mechanism enables insects to detect VOCs with high selectivity and sensitivity [23-25].

M. atrata approaches eucalypt forest stands shortly after the fire as the first colonizer to avoid competition and predators. The beetles mate and oviposition selected eucalypt stems [26]. They use their IR sensitive organs, which are situated on the ventrolateral sides of the abdomen, to detect fires and to avoid landing on hot patches [26-29]. This study investigates the temperature dependent VOC emissions of *E. globulus* wood and leaves. The olfaction of the fire beetle *M. atrata* is used to learn from nature's evolutional adaption to fire detection. The resulting concept is transferred to semi-conductor gas sensors, which are used to detect an increasing fire risk.

Material and Methods

Thermal Oxidation of Wood Chips, Leaves, and Wood Chips with Leaves of *Eucalyptus globulus*

Wood and leave samples of five *E. globulus* trees were cut into 1mm flakes and 0.5 cm² pieces, respectively. 2g of each material was used for the experiments with wood and with leaves. For the experiments with both wood and leaves, 1g wood chips and 1g leaves were mixed. A reconstructed GC oven (Fractovap Series 4160, Carlo ErbaStrumentazione, Rodano, Italy) was used to heat

the *E. globulus* samples. Synthetic air (1bar, 20% O₂ in N, Alpha Gaz, Düsseldorf, Germany) was led into a glass flask that contained the sample. A rotary vane pump (Thomas Division, Sheboygan, USA) sucked 0.6 L air in 30 s out of the flask through calibrated charcoal filters (1,8 mg Charcoal, CLSA Filter, Daumazan sur Arize, France) to take an empty control sample and samples at 25°C, 50°C, 75°C, 100°C, 150°C, 200°C, 250°C, and 300°C.

VOC Analysis and Quantification

The charcoal filters were washed with 75 µl dichloromethane/methanol (2:1). 1µl of the eluate was analyzed by a GC-MS system (6890N Network GC System, 30 m × 0.25 mm HP-5 MS, 95% Dimethyl- 5% Diphenyl-Polysiloxane, nonpolar column with 0.25-µm film thickness / 5973 quadrupole mass spectrometer, Agilent Technologies, Santa Clara, USA). The following oven program and detector parameters were applied: initial temperature: 40°C for 2.5 min, rate 1: 6, 2°C per min to 250°C, final hold: 10 min, MS in scan mode (m/z detection range of 20-354 per 0.5 s). The VOCs were analyzed with the Enhanced Chemstation Version D00.00.38 software (Agilent Technologies, Santa Clara, USA), the Mass Spectral Search Library of the National Institute of Standards and Technology NIST (Gaithersburg, USA), and the MS Interpreter Version 0.9f (Gaithersburg, USA). The identity of VOCs was further confirmed by calculating the linear retention index (C7 to C24 straight-chain hydrocarbons [30]) by using purchased standard VOCs at 10⁴ dilution (Table 1). The area of the most abundant ion in the purchased standard VOC MS-spectra was used for quantification. The emission rate was calculated in ng/L×60s. This raw data was analyzed for normal distribution by the Shapiro-Wilk test, and, as most data sets were not normally distributed, displayed in box plots showing the median, upper/lower quartile, minimum, and maximum of the respective emission rates. Table 1 shows the identified VOCs of *E. globulus* leaves, wood or wood and leaves in the order of their retention index within the respective chemical group on a nonpolar HP-5 column.

VOC	Linear retention index (LRI)	CAS No. of purchased standard VOCs	Vapor pressure of pure VOC at SATP [mmHg]
Terpenes			
α-Pinene	937	80-56-8	4.75
Camphene	952	79-92-5	3
β-Pinene	980	127-91-3	2.93
β-Myrcene	993	123-35-3	2.29
Limonene	1037	138-86-3	1.55
Eucalyptol	1042	470-82-6	1.9

Camphor	1162	464-49-3	4
Aromadendrene	1450	72747-25-2	0.02
Epiglobulol	1583	88728-58-9	0.03
Furanes			
Furfural	860	98-01-1	2.234
2-Furanmethanol	877	98-00-0	1.009
5H-Furan-2-one	950	497-23-4	0.273
5-Methylfurfural	975	620-02-0	0.644
5-Hydroxymethylfurfural	1245	67-47-0	0.001
Methoxyphenols			
2-Methoxyphenol	1105	90-05-1	0.179
4-Methyl-2-methoxyphenol	1209	93-51-6	0.078
4-Ethyl-2-methoxyphenol	1296	2785-89-9	0.017
2-Methoxy-4-(2-propenyl)phenol	1375	97-53-0	0.01
2-Methoxy-4-formylphenol	1421	121-33-5	0.002
Benzenes			
1-Isopropyl-4-methylbenzene	1031	99-87-6	1.46

Table 1: The identified and quantified VOCs from the wood and leave, leave, and wood samples. The VOCs are assigned to their respective chemical group and, within this group, listed in the order of their linear retention index. The CAS number of the purchased standard VOC is given for exact identification and the vapor pressure of the VOCs for interpreting the results of the EAG are given, as well.

Insects and EAG

In summer 2009 and 2010, adult *Merimna atrata* were collected one to ten days after a fire on stems of charred eucalypt trees in the Dwellingup State Forest, Western Australia, in the Mundaring State Forest, Western Australia, and in summer 2010 and 2011 in the region of Jurien Bay, Western Australia. They were kept on 10% saccharose sugar water and water diet at $25 \pm 1^\circ\text{C}$ / 40% rel. hum. and a 12/12 day/night period. Before

performing the electro-antennogram serial dilutions of the VOCs (EAG), the electroantennographic (EAD) detection was performed (data not shown, method described in [13]) in order to select electrophysiological active VOCs. These VOCs (Table 1) were diluted with paraffin oil (Uvasol quality, Merck/VWR, Darmstadt, Germany) at different concentrations (10^{-7} to 10^{-2} g/g). App. 200 μL of the mixtures were dropped on 2-cm² filter papers and put into a 10-ml glass syringe. 2.5 ml of the airspace in the syringe was led into a stream of humidified air (500 ml / min, 23°C, 80% rel. hum.) and directed to the excised antenna of the beetles. The signal output was mV \times 100 (amplification factor). The procedure was repeated three times per dilution step and beetle (n=3) and on five animals (n=5 for the species). For more technical details on the EAG setup, see [31]. According to the results of the Shapiro-Wilks test for normal distribution, the T-test for paired samples or the Mann-Whitney U-test was applied to test for significant differences between mixture and paraffin oil controls.

Gas Sensors

Three gas sensors GGS5330 WO₂, GGS2330 SnO₂, and GGS1330 SnO₂ (UST-Umweltsensortechnik, Geschwenda, Germany) were operated at 200 Ω , 200 Ω , and 75 Ω , respectively, and at 5 V. The VOCs emitted during thermal oxidation experiments of *E. globulus* wood, leaves and wood and leaves with temperatures from 25°C to 240°C were led through a sensor chamber and the resistance of the sensors was measured in intervals of 1s. The sensors were calibrated on eucalyptol and furfural and during the thermal oxidation samples VOC samples were taken at 25°C, 150°C, 200°C, and 240°C and analyzed as described above. The same setup was used as for the VOC sampling, but instead of the glass cylinder, a stainless-steel cylinder with lower heat conduction was used. This decelerated the heating of the biomass and simulated the pre-ignition situation. The biomass was not burned but showed a dark brown color after the sensor experiments.

Results and Discussion

Nine terpenes, five furans, five methoxyphenols, and one benzene compound were identified and quantified in the VOC analysis. The vapor pressures of the compounds corresponded to the order of their linear retention indices (Table 1). Camphor was the only exception (Table 1). The VOC emission pattern showed clear qualitative and quantitative trends (Figure 1).

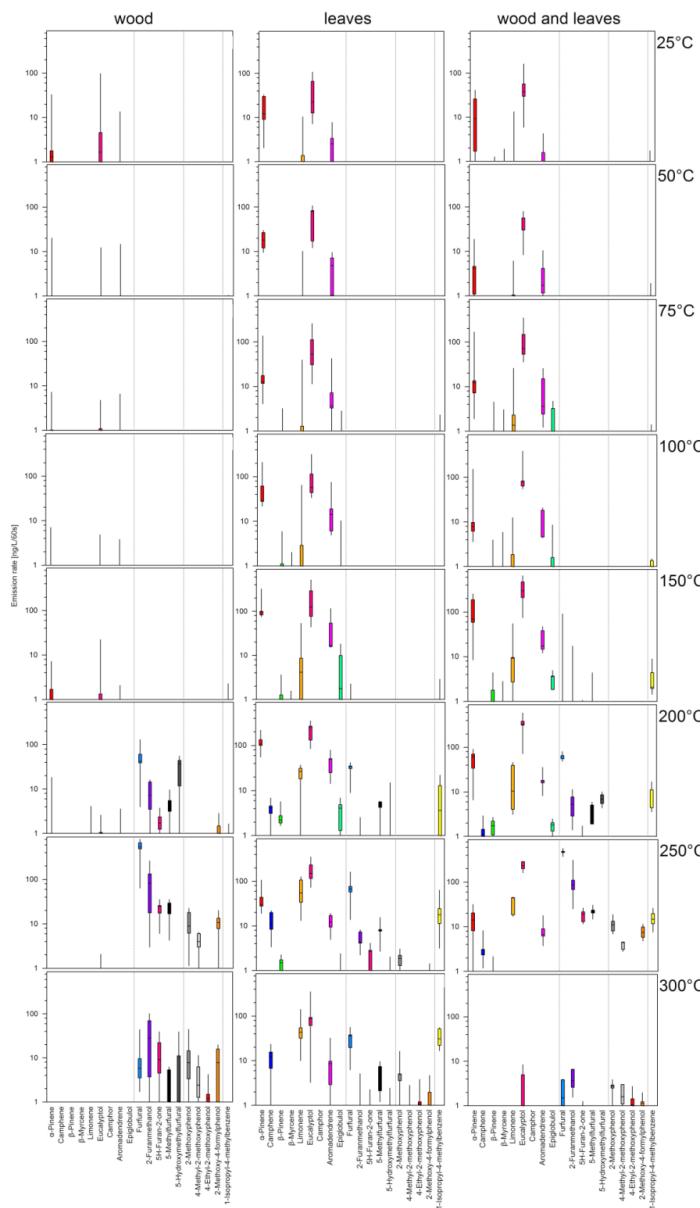


Figure 1: Emission rates of the eucalypt wood, leaves, and wood and leaves in ng/L×60s on a logarithmic scale for each temperature step between 25°C and 300°C. For all VOC data sets, the median, the upper and lower quartile, the maximum and the minimum are displayed in box plots. The dotted lines group the VOCs in terpenes, furans, methoxyphenols and the benzene compound

In all three tissues the ignition took place in the heating phase from 250°C to 300°C. This is in accordance with the auto-ignition temperature of solid wood at low heat fluxes without direct exposure to a flame [32]. The VOC emission rates in the samples taken at 300°C were low, as only ashes and residual VOCs had remained in the glass flask at this temperature. The terpenes

were mainly emitted by the leaves, while wood contained low amounts of α -pinene and eucalyptol, as well. The furans and the methoxyphenols were mainly emitted by the wood as products of the thermal degradation of hemicellulose/cellulose and lignin, respectively [13]. Furans and methoxyphenols were also emitted by the leave tissue, as the leaves contained low amounts of lignocellulose, as well. In all three tissues the terpenes were already emitted at low temperature. Their emission rate was correlated to the temperature (Clausius Clapeyron equation) and to the concentration in the tissue (Henrie's law or Raoult's law). Within the group of terpenes, the bicyclic monoterpenes α -pinene and eucalyptol had the highest emission rates in all three tissues (Figure 1). At the temperature stages between 25°C to 150°C both VOCs constituted 87-96% (standard deviation 3-10%) of the total emission from the wood and leaves samples. The relative amount of eucalyptol and α -pinene in the total emissions decreased from 70% (12% standard deviation) at 200°C to 26% (5% standard deviation) at 250°C and 11% (12% standard deviation) at 300°C. Eucalyptol is the only VOC that was emitted throughout the heating process in the wood and leaves samples with an emission maximum between 150°C and 250°C (Figure 1).

Limonene, β -pinene, epiglobulol, and aromadendrene were emitted by the leaves (Figure 1) with a comparable temperature related dynamic as eucalyptol and α -pinen. The terpenes camphor, β -myrcen, and camphene did not show this dynamic. It is possible that camphene had formed due to oxidation of α -pinene at high temperature, as it is suggested by Neuenschwander et al. [33]. This would explain its exclusive presence at 200°C and 250°C in the leave and in the wood and leaves samples. Camphor occurs at very low emission rates in the leaves samples at 300°C which suggests that it is an oxidation product of camphene, which was the only structurally related terpene present at this temperature (Figure 1).

In the wood, leave and in the wood and leaves samples the thermal degradation started of hemicellulose, and/or cellulose started at 150°C. At 200°C all leaves samples emitted furfural at emission rates between 9 and 42 ng/L×60s (7% total emission, 3% standard deviation). All wood and leaves samples emitted furfural at emission rates between 49 and 81 ng/L×60s (15% total emission, 8% standard deviation). The wood samples showed furfural emission rates in the range of 4 to 129 ng/L×60s (73% total emission, 10% standard deviation)

The furfural emission of all wood and leaves samples increased further to 250°C (50% total emission, 8% standard deviation) and decreased to 300°C (11% total emission, 5% standard deviation). At 250°C the sample 2 of the wood and leaves samples showed the highest emission of a single VOC in the whole emission pattern of the wood and leaves samples (810 ng/L×60s, 68%, and the group of furans had the highest total emission among the four chemical groups. All wood and leaves samples emitted

lower amounts of secondary products of furfural (2-furanmethanol, 5-methylfurfural, and 5-hydroxymethylfurfural). All three tissues emitted methoxyphenols at 250°C and 300°C. The emissions of methoxyphenols from the wood and leaves samples were lower than the emission of terpenes and furans and took place exclusively at 250°C and 300°C. Within this group, 2-methoxyphenol showed the highest emission followed by 2-methoxy-4-formylphenol. 1% (0.3% standard deviation) of the total emission at 250°C and 12% (8% standard deviation) at 300°C were 2-methoxyphenol and 2-methoxy-4-formylphenol.

The antenna of *M. atrata* responded to most of the terpenes. The intensity of the reaction in mV is correlated to the vapor pressure of the terpene. A higher vapor pressure leads to a stronger reaction. Terpene emissions are not relevant for fire detection, as the risk of false alarm scenarios for the beetle is very high. In a forest ecosystem there are many sources of terpenes, e.g. trees under drought stress, physically damaged conifers, and flowers. The antennae responded significantly to furfural and 2-furanmethanol at a dilution of 10⁻⁴ (Figure 2 and 3). Other furans do not elicit an electrophysiological response, as they have a lower vapor pressure (Table 1). Furan indicates the start of the degradation of wood, which can be a cue for the beetle. The VOC with the best relation of vapor pressure (0.179, Table 1) and detection limit (10⁻⁴, Figure 2 and 3) is 2-methoxyphenol. This VOC is the most sensitive detected in this study, as its vapor pressure is low, and the detection limit is low, as well. The ecological importance of 2-methoxyphenol is also suggested by another study on a related fire beetle species [34]. The beetle is able to sense furans and 2-methoxyphenol, which enables it to detect smoke plumes and lignocelluloses at high temperatures.

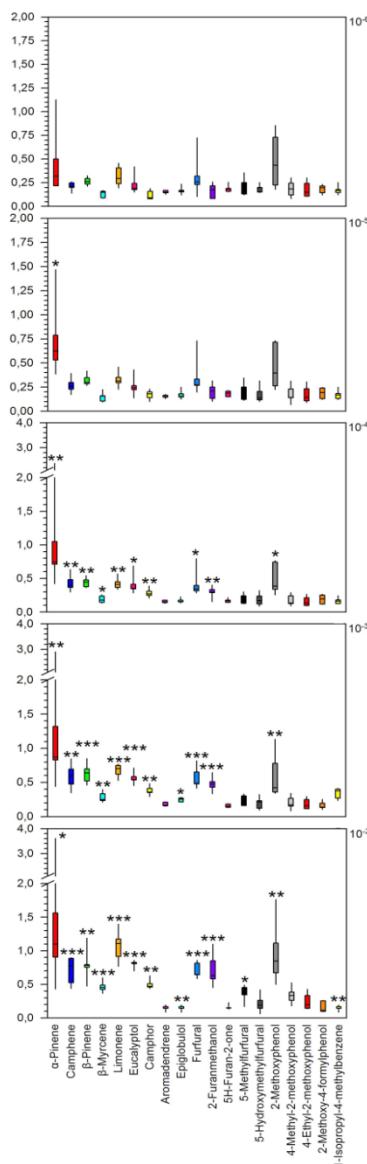
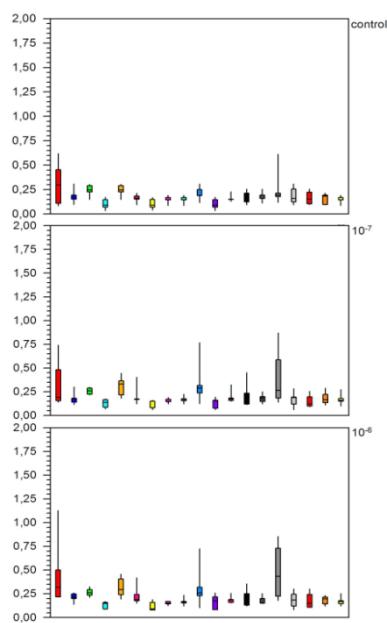


Figure 2: Results of the electrophysiological studies on the antenna of *M. atrata*. The antennal responses are given in mV and data sets (N=5) are displayed showing the median, the upper and lower quartiles and the minimum and maximum in box plots. Significant differences between the paraffin control and the respective VOC dilution were calculated based on the Mann-Whitney U-test or the t-test and displayed with stars (*: p<0.05; **: p<0.01; ***: p<0.001).

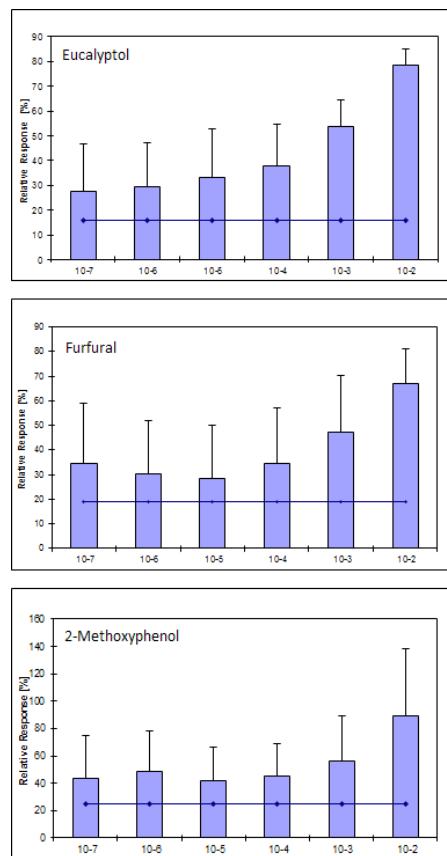


Figure 3: Dose response curves of *M. atrata* for eucalyptol, furfural and 2-methoxyphenol. The relative response in % was calculated on the basis of an individual standard stimulus.

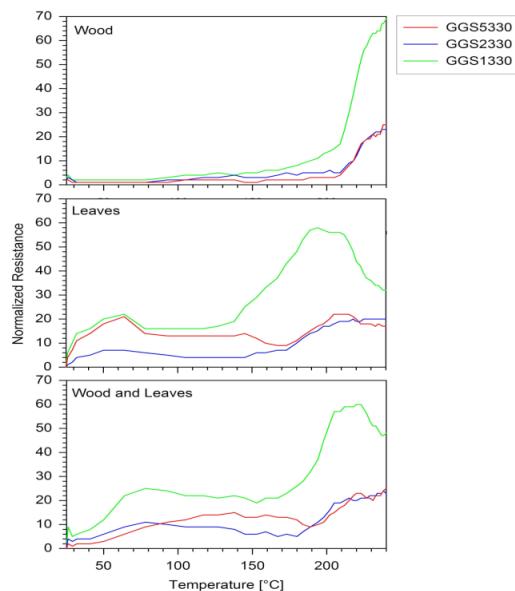


Figure 4: Results of the sensor experiment. The three sensors

can detect the two stages of the thermal treatment of *E. globulus* biomass before ignition, which are the terpene emission phase (25-150°C; GGS5330 and GGS1330) and the onset of furan emission from degradation of lignocellulose (150-200°C; GGS1330). This sensor setup allows the detection of heated *E. globulus* biomass before it ignites and, therefore, can serve as an early fire warning system to prevent the ignition of a fire or to allow a fast response of fireman.

Figure 4 shows the temperature dependent response of three selected semi-conductor metal oxide gas sensors to the VOCs emitted during the thermal treatment of *E. globulus* biomass.

As furan emissions are tree species unspecific, a sensor system detecting furfural could assess the fire risk of any lignocellulose biomass. Sources of false alarm can be microbial or auto-oxidative degradation of dead wood and dry litter, fungal infection of dead wood or cooked meat [35]. However, the kinetics of thermally induced oxidative depolymerization is faster than auto-oxidative or enzymatic processes. Therefore, the emission rate of furfural or furans from other sources is low. Furfural is not emitted by decaying vertebrates [36,37].

The onset of a fire is not necessarily gradual, but can be spontaneously, e.g. caused by hot exhaust fumes of motorbikes or burning cigarettes. A fire risk assessment before the ignition is not possible in such cases. Another restriction of the proposed sensor system is the uneven and unpredictable spatial distribution of odor plumes. This could result in a dense network of sensors or in a concentration of a few sensors at hot spots, e.g. for the protection of households. A sensor system in eucalypt plantations or forests can include methoxyphenol detection to further increase its operating spectrum from fire risk assessment by furan emission to smoke detection. As methoxyphenols are, like furans, species unspecific, this feature can be applied for any lignocellulose biomass.

Conclusion and Outlook

The results of the VOC analysis showed that there is a temperature dependent VOC profile of *E. globulus* biomass. The antennae of the fire beetle *M. atrata* can detect the key components in heated biomass in order to approach a woodland fire. The proposed sensor setup can detect these key components and allows an early fire warning in plantations or forests. Further research has to be conducted on the performance of the sensor system under field conditions.

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