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Investigating PM10 episodes using levoglucosan as tracer

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Abstract The present study aims to investigate the role/contribution of residential combustion (using levoglucosan as a tracer of biomass combustion) during PM10 episode days registered over the Porto urban area (Portugal), in order to support air quality plans that need to be developed for this particular region. The levoglucosan and PM10 concentration values, together with the meteorological conditions (namely temperature), measured during an experimental field campaign performed in 2013, were used in this study. To this end, a wavelet-based approach is applied to (a) better quantify the coherence and dependency of these variables and (b) assess the strength of the connection between the two pollutants species (PM and levoglucosan) at different time scales. Results evidenced a high coherence/dependency between PM10 and levoglucosan values for the episodes selected (periods with exceedances of the PM10 limit values), suggesting the contribution of biomass combustion sources. The highest coherence (normalised covariance) is observed for the winter episodes and time periods of 5–10 days, which is related to the duration of the episodes selected. The summertime episode, which exhibits a negligible observed correlation between temperature and levoglucosan, is explained by the influence of forest fires that occurred within this period and region.

Keywords Levoglucosan · Temperature · Particulate matter · Episodes · Wavelet analysis

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Introduction

Air quality is one of the environmental areas in which the European Union (EU) has been most active, in particular designing and implementing legislation and setting limits and target values for concentrations of major air pollutants (Air Quality Directive 2008/50/EC). This directive reinforces the obligation of EU member states to elaborate and implement air quality plans (AQP) to improve air quality when standards are not fulfilled. The implementation of AQP, when pollutant concentrations exceed the air quality standards in zones or agglomerations, should be based on the development of measures that reduce the pollutant atmospheric concentrations and meet the legal requirements (Miranda et al. 2015).

Exceedances of the thresholds of particulate matter (PM10) have been reported in the urban area of Porto (Portugal), where human exposure is also high (Monteiro et al. 2007; Borrego et al. 2009; Miranda et al. 2015). Air quality plans were already developed for this pollutant during the period 2005–2008 for PM10 (Borrego et al. 2010). Besides improvements in air quality, verified after this period, a reduction in the concentration of this pollutant is still required because legislated limits continue to be surpassed every year in specific monitoring sites (Duque et al. 2016).

Residential wood combustion is of increasing concern as it is identified as a major source of atmospheric pollution, mainly in winter (Gelencsér et al. 2007; Krecl et al. 2008; Lanz et al. 2007; Puxbaum et al. 2007; Reche et al. 2012). Although

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its use is recommended, given that it reduces the dependency on fossil fuels, it is responsible for large impacts on air quality, as well as potential effects on public health and global climate change (Bølling et al. 2009; Vu et al. 2012). Modelling results indicated that in Portugal 18% of PM10 could be related to this emission source (Borrego et al. 2010). Source apportionment results obtained from a PM2.5 (PM less than 2.5 μm in aerodynamic diameter) database obtained in a coastal/rural area in Portugal showed that 52 to 69% of the organic carbon can be attributed to residential wood combustion (Gelencsér et al. 2007).

A wide range or variability has been found for particulate matter (PM) emissions. This variability has also been observed for levoglucosan ($C_6H_{10}O_5$, formed from the pyrolysis of carbohydrates, such as cellulose), commonly used as a molecular marker for biomass burning (Hedberg et al. 2002; Gonçalves et al. 2012; Vicente et al. 2015).

The present study aims to investigate how levels of levoglucosan and meteorology (temperature) conditions are associated with PM10 (particulate matter less than 10 μm in aerodynamic diameter) episodes registered in a Portuguese urban area, in order to investigate the role/contribution of residential combustion. Experimental data from a field campaign performed in Porto throughout 2013 were used. These PM10 episodes were selected because of the exceedances of the legislated values. A statistical method based on a wavelet approach (Monteiro et al. 2016) was applied to better quantify the coherence and dependency of these 3 variables. Since air pollutant time series are of non-stationary nature with substantial temporal changes in periodicities of environmental interest (e.g. a daily period), the link between these variables is explored by wavelet transforms. In particular, wavelet coherence and phase were used to clarify the strength of the connection between both pollutants species (PM and levoglucosan) at different time scales. Special attention was given to the coherence relationship found for the PM10 episodes registered during the field campaign period.

The present work is organised as follows: the 'Data collection and analysis' section presents the monitoring data used in this study, namely PM10 concentration values, levoglucosan and also the correspondent meteorological data. A statistical analysis using a wavelet-based approach trying to correlate these set of variables is performed in the 'Statistical method' section. Finally, conclusions are drawn in the 'Summary and conclusion' section.

Data collection and analysis

Field campaign and levoglucosan levels

Sampling was carried out within the frame of the AIRUSE project (Amato et al. 2016), which aimed at characterising the main sources contributing to the particulate matter levels in Southern European countries. A combination of multiple emission sources with complex climatology (strong radiation, high photochemical conversion rates, low rainfall rate) leads to frequent exceedances of the limit values. The sampling equipment was installed on the roof of the station located at Praça Francisco Sá Carneiro (41°09′46.10″N, 8°35′26.95″W), in the city of Porto. The station belongs to the Portuguese air quality monitoring network and is classified as 'traffic'. A high-volume sampler operating at a flow of 1113 L min^{-1} with impaction plates from Sierra-Anderson was used to collect PM2.5 onto quartz fibre filters. Samples were collected from January 2013 to January 2014 every third day.

Levoglucosan was determined by high-performance anion-exchange chromatography with pulsed amperometric detection (HPAE-PAD). The chromatograph (model 881, Metrohm, Switzerland) was equipped with an auto sampler Methrom, an amperometric detector for levoglucosan (module 896, PAD mode, Metrohm, Switzerland), and a column Metrosept carb 1 (150 mm × 4 mm id). Particulate matter on each filter punch (1.5 cm^2) was extracted with Milli-Q water (10 mL) through ultrasonic agitation. After extraction, the solution was filtrated through a polypropylene puradisc (0.45 μm porosity, 25 mm diameter). The detection limit of the method was 0.075 μg cm^{-2}.

The levoglucosan mean level for the whole campaign was 407 ng m^{-3}, whilst a winter mean value of 774 ng m^{-3} was registered. These values are possibly underestimated because levoglucosan can be degraded in the atmosphere, especially oxidised by OH radicals, as reported in some simulation experiments and model studies (Hennigan et al. 2010; Hoffmann et al. 2010). It should be noted that some peak events were detected during the summer as a consequence of the impact of emissions from wildfires and/or agricultural fires, although the sinter mean value is higher than in the summer. Concentrations of levoglucosan in PM2.5 samples from Porto are in the range of values reported for other urban sites in the USA (e.g. Brown et al. 2016; Nolte et al. 2001; Zhang et al. 2010) and Europe (e.g. Caseiro et al. 2009; Giannoni et al. 2012; Perrone et al. 2012; Piazzalunga et al. 2010; Zdráhal et al. 2002) and higher than those measured in mountain and oceanic background atmospheres (e.g. Mochida et al. 2010; Puxbaum et al. 2007).

The application of Positive Matrix Factorisation (PMF) to the chemical databases obtained in the AIRUSE project (Amato et al. 2016) revealed that, on average, biomass accounted for 12% (4.2 μg m^{-3}) and 18% (4.4 μg m^{-3}) of the PM10 and PM2.5 levels, respectively. The impact of biomass burning emissions was especially high in the winter months, due to the generalised use of wood for residential heating (Gonçalves et al. 2012). The contribution of biomass burning to PM was also higher in September. During this particularly hot and dry month, several wildfires were

Fig. 1 Sampling station in Porto where the AIRUSE monitoring campaign took place

registered in the Porto district. According to the Institute for Nature Conservation and Forests (ICNF, 2014), this district recorded the highest number of occurrences and one of the largest burnt areas. When the mean source contributions that have been estimated by PMF with the annual dataset are compared with the assignments for days in which the PM10 levels exceeded the limit of 50 μg m^{-3}, a salient feature caught the eye. The percentage contribution from biomass burning to PM10 and PM2.5 doubled on exceedance days: 16.5 μ m^{-3} (25%) and 23.6 μg m^{-3} (37%), respectively (Amato et al. 2016). It is worth noting that approximately half of the exceedances were recorded in late August and early September, when intense wildfires struck the region. The other half was registered in winter months, suggesting the input from residential wood combustion to the atmospheric particulate loads Fig. 1.

Air quality and temperature data

The time series of PM10, levoglucosan and temperature, measured in the field campaign, are plotted in Fig. 2. Temperature data exhibit an annual time profile with seasonal variation, with thermal amplitude between 2 and 18 °C during winter and between 10 and 30 °C during the summer period. The PM10 hourly time series have an annual average of 24.5 μg m^{-3}, with minimum values below 10 μg m^{-3}, during periods of strong precipitation not coinciding with rush hours, and maximum peaking above 100 μg m^{-3}.

Episodes selected

In order to analyse the periods in which PM10 registered very high levels, above the limit value for protection of human

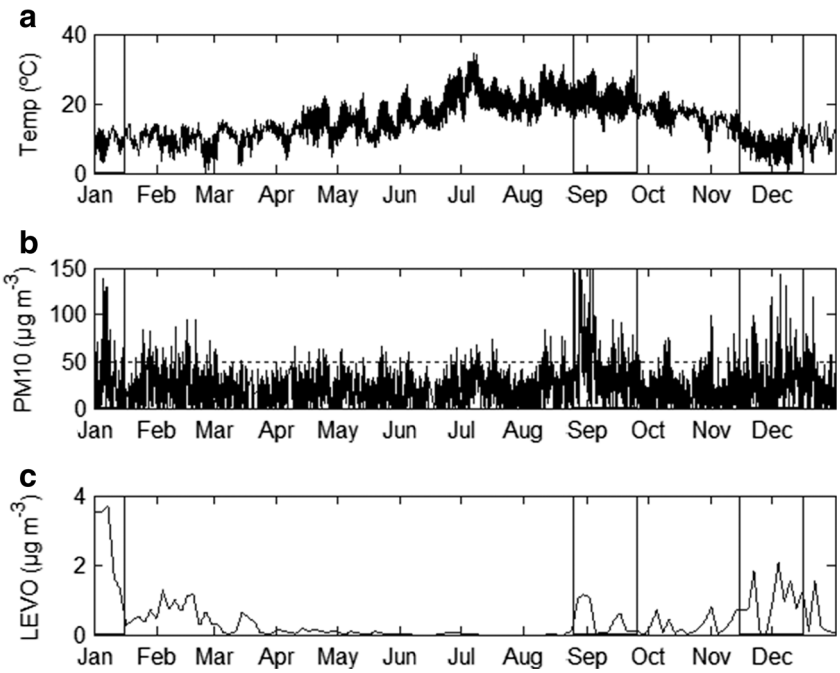

Fig. 2 Temperature (**a**), PM10 concentrations (**b**) and levoglucosan (**c**) data measured during the 2013 field campaign. Temperature (1 observation per 6 h) and levoglucosan (1 observation per 3 days) were linearly interpolated to the same temporal scale as that of the PM10 (1 observation per hour). Vertical thick lines delimit the three selected episodes

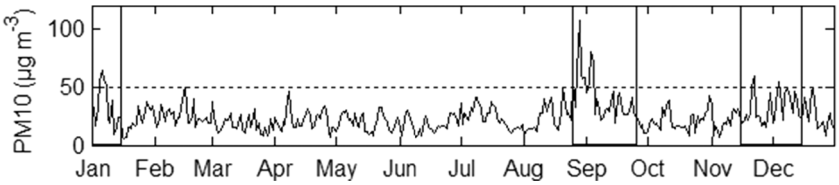

Fig. 3 Daily average of PM10 concentrations. Vertical thick lines delimit the three selected episodes

health defined by legislation (50 μg m^{-3} for daily average), the daily average time series for PM10 was computed, as shown in Fig. 3.

From Fig. 3, it is possible to identify 3 distinct periods with values above the limit value for protection of human health, 2 of them during the winter season and 1 during the summer: 1st–15th January, 25th August–25th September and 15th November–15th December.

Figure 4 shows the three variables analysed—PM10, levoglucosan and temperature—observed for each episode selected, allowing a better characterisation of these time periods.

The PM10 episodes registered during the winter period (1st and 3rd) are characterised by low-temperature values (minimum below 5 °C and maximum of 15 °C). Both episodes exhibit several large peaks of PM10 and high levels of levoglucosan. The episode observed during summertime (August–September) has high values of temperature (maxima around 35 °C), but lower levoglucosan values when compared to the other episodes.

Statistical methods

The temperature and levoglucosan time series were resampled for an hourly temporal basis (8760 observations = 24 observations/day × 365 days) by linear interpolation. Next, the dependence structure between pairs of series was explored by the wavelet transform (WT) decomposition through the computation of the coherence and the phase lag between the time series as a function of time and of frequency (Percival and Walden 2006; Zeri et al. 2011; Zeri et al. 2016).

Let X_t represent the input and Y_t the output a system, both defined for $t = 1, \ldots, T$. The cross-wavelet transform between the two series is defined as

$$W_{xy}(j,t) = W_x(j,t)W_y^*(j,t)$$

where $W_{xy}(j, t)$ is the wavelet transform of X_t and $W_y^*(j,t)$ is the complex conjugate of $W_y(j, t)$. The index $j = 1,2,\ldots J$ relates to the wavelet scale 2^(j − 1) that captures the joint dynamics of the series over intervals with duration from 2^j to 2^(j + 1) time units.

The covariance between X_t and Y_t was described in terms of the normalised squared modulus of *Wxy(j, t)*, i.e. the squared coherence, defined as

$$C_{xy}(j,t) := \left|W_{xy}(j,t)\right|^2 / W_x(j,t)W_y(j,t),$$

that corresponds to a real valued function varying between 0 and 1, where 1 indicates a perfect linear relationship between the two series at a given time t and a given frequency scale j. In addition, phase delay between X_t and Y_t was obtained from the complex argument of ,

$$W_{xy}(j,t),$$

where a negative value indicates that Y_t leads X_t whereas a positive value indicates that X_t leads Y_t.

Squared coherence and phase were estimated using a Morlet wavelet with dimensionless frequency $w_0 = 6$, since

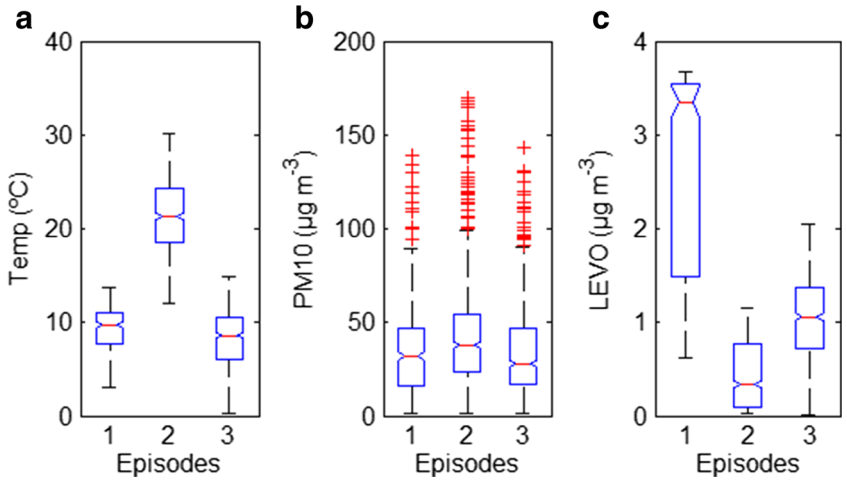

Fig. 4 PM10, Levoglucosan and temperature data registered during the—3 episodes selected: (1) January; (2) Aug-Sept and (3) December

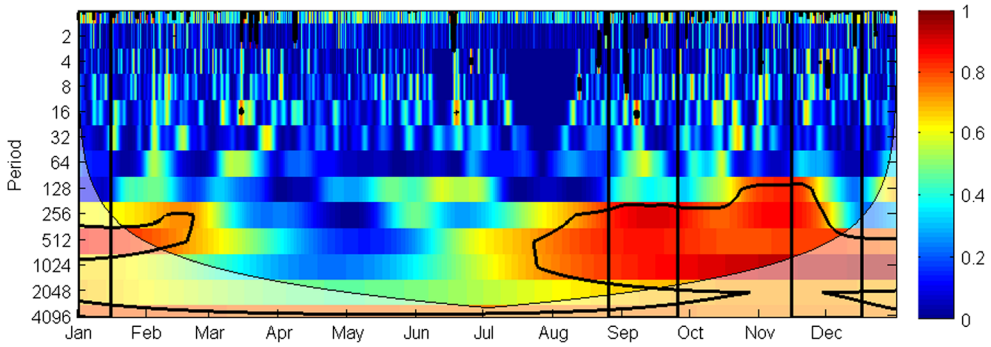

Fig. 5 Squared wavelet coherence showing the strength of linear relation between PM10 (input) and levoglucosan (output) as a function of time and frequency. The shaded areas delimit the results influenced by edge effects of the wavelet analysis. Areas with 5% significance level against red noise are shown as a thick contour. Vertical thick lines delimit the three selected episodes: 1st–15th January, 25th August–25th September and 15th November–15th December

it provides a good trade-off between time and frequency WT resolution (Grinsted et al. 2004). Furthermore, $J = 12$ was set to obtain the wavelet scales with period 1, 2, 4, … 4096 thus including several periodicities in the WT analysis. The statistical significance of the coherence (and consequently the phase reliability) was assessed by simulation, described as follows (Torrence and Compo, 1998, Grinsted et al. 2004): an ensemble of 300 (stationary first-order autoregressive) surrogate time series was generated from the original *Xt* and *Yt* time series, being the wavelet coherence estimated for each pair of surrogates. The significance level was set as the 95%-percentile of the surrogate coherence amplitudes, for each WT scale $j = 1,\ldots, J$. Coherence values in the original series observed above this threshold were considered significant (at a 5%-level).

Results and discussion

Figure 5 shows the cross-wavelet transform/coherence of PM10 and levoglucosan time series for the entire 2013 year, showing the strength of linear relation between PM10 and levoglucosan as a function of time and frequency and highlighting the three selected episodes.

All episodes exhibit a high coherence/dependency between PM10 and levoglucosan values thus, suggesting that, on these time intervals, biomass combustion sources exist and contribute to these peaks occurrence. Furthermore, during March–June period, there is no significant coherence between PM10 and levoglucosan values thus indicating no linear dependency between PM10 and levoglucosan for all frequency scales.

Figure 6 shows the phase between PM10 (input) and levoglucosan (output) as a function of time and frequency, highlighting the positive (red pallet colours, PM leads levoglucosan) and the negative (blue pallet colours, levoglucosan leads PM) values. It is important to stress that it is not possible to estimate time delays lower than 1 day with a sampling frequency of one observation per day. As air quality and temperature data are known to exhibit intra-daily variations, the phase analysis focused the sign (positive or negative) which provides an interpretation on how the input and output of a linear system synchronise to achieve highest coherence.

For episode 3, the positive values period of the phase indicate that the highest coherence values are obtained for positive phase values, thus suggesting that PM10 leads levoglucosan values in this time interval. The same pattern is observed for episodes 1 and 2, except for the wavelet scales 512 and 1024 (episode 1) and wavelet scale 1024 (episode 2), where negative phase values between PM10 and levoglucosan are observed. The frequency scales 512 and 1024 correspond to the analysis of time intervals with duration between 43 and 85 days and between 85 and 171 days, respectively. The different (positive and negative) values of the phases found can be justified by the different sources (PM is not emitted only by

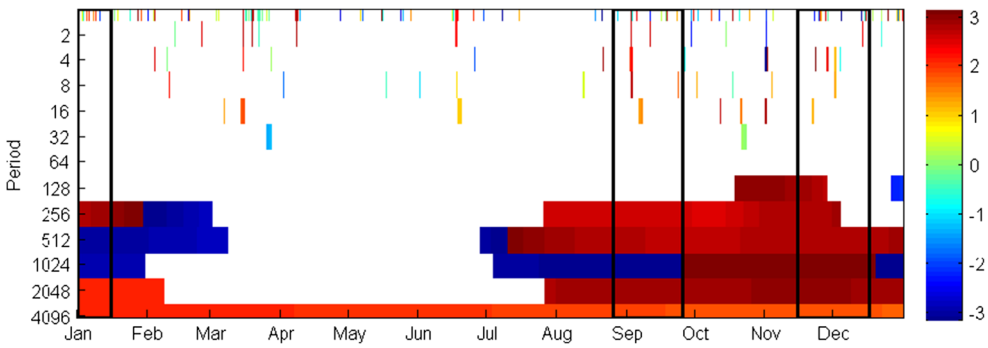

Fig. 6 Phase delay between PM10 (input) and levoglucosan (output) as a function of time and frequency, highlighting the positive (red pallet colours, input leads output) and the negative (blue pallet colours, output leads input) values. The green colour identifies those cases in which the phase between the series is close to zero. Vertical thick lines delimit the three selected episodes

biomass combustion sources) and dispersion phenomena (different weight/mass) that exist between levoglucosan and PM10.

The same analysis was performed for temperature and levoglucosan time series. The wavelet transform for the temperature and levoglucosan time series (temperature > levoglucosan) and the respective phase between these two variables are shown in Fig. 7.

As expected, there is higher dependency between levoglucosan and PM10 than with temperature, since levoglucosan is part of particulate matter compounds.

The highest coherence between levoglucosan and temperature is found for the winter episodes and for time periods of 5–10 days, which is related to the duration of the episodes (period = 64). This time scale of a few days suggests a potential relationship with the weather/synoptic conditions, namely the passage of frontal systems which introduce a variability of 3–5 days on variables such as air temperature (and pollutants concentrations as a consequence), as supported by previous studies (e.g. Zeri et al. 2011, 2016).

This high dependency found suggests that biomass combustion is an emission source responsible for these PM10 high values, which during winter season is mainly associated with residential combustion (use of domestic fireplaces and woodstoves) (Calvo et al. 2015). For the summertime episode (August–September), no relevant correlation was observed for all time periods. This can be explained by the origin of this particular episode, which was mainly influenced by forest fire occurrences (JRC 2014). Regarding the phase analysis (Fig. 7b), no significant delay is found between these two variables since phase values are close to zero.

Summary and conclusions

This study intends to characterise how levoglucosan concentration measures are correlated to PM10 and to temperature values. Since levoglucosan is a tracer of biomass combustion, these correlation results will help identify the origin of PM10 episodes registered in Porto urban area. Data were measured during an experimental field campaign in 2013, and the dependence and phase between the two pollutants species (PM and levoglucosan) were analysed at different time scales through wavelet transform. Results pointed out a high dependency between PM10 and levoglucosan values for the winter episodes selected suggesting a strong contribution of biomass combustion sources for the exceedances of PM10 daily limit values. The summertime episode, with no relevant correlation observed between temperature and levoglucosan, is explained by the influence of forest fires that occurred in this period and region. This statistical analysis is particularly important to investigate the origin of the PM10 episodes registered in Porto urban area each year, especially the contribution of residential biomass combustion in winter. The fact that wintertime variability of air temperature has a higher impact on pollutants will be very important to establish efficient measures to reduce this pollutant concentration, in particular to

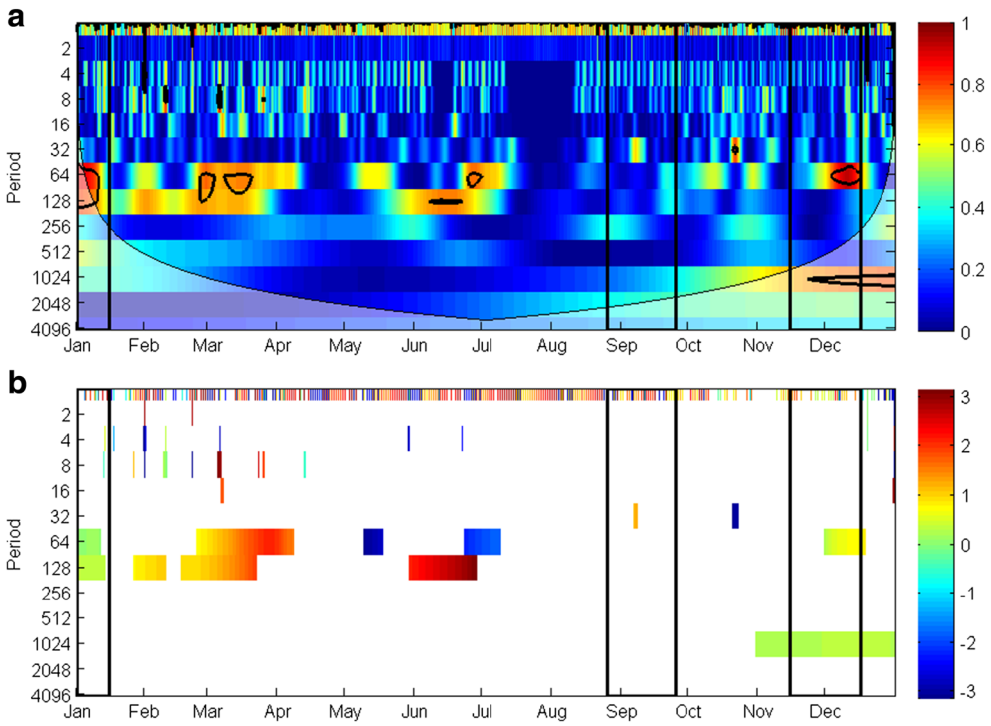

Fig. 7 **a** Squared wavelet coherence and (**b**) phase delay between Temperature (input) and levoglucosan (output) as a function of time and frequency. Same colour scheme and representations as in Figs. 5 and 6. Vertical thick lines delimit the three selected episodes

support the air quality plans that need to be developed for this specific urban area.

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