DESIGN, CONSTRUCTION AND TESTING OF A LABORATORY CHAMBER FOR DUST GENERATION AND SAMPLING

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EXTENDED ABSTRACT
The first step in addressing the atmospheric pollution problem faced by industrial and urban areas is the creation of a pollution sources inventory followed by the quantification of each source contribution to the measured pollutants concentrations. The reliability of the applied method affects directly and decisively the effectiveness of the subsequently adopted measures towards the air quality management.

There is a variety of methods globally applied in order to link the air pollution sources to the measured pollution concentrations. The most widespread one, is known as «receptor modeling» and comprises of sampling in the study area by creating a network of proper instruments around the area, chemical analysis of samples and finally of the calculation of each source contribution to the air pollution of the area. The main disadvantage of this method is the difficulty in isolating the contribution of each source to the environmental burden of the receptor area.

The problem is more complex in the case of particulate matter with aerodynamic diameter less than 10 μm (PM10) due to the variety of emission sources linked to particulate matter e.g. burning of biomass, industrial stacks, unpaved roads, and other fugitive sources. In order for any air quality management plan to be applied it is of great importance to attribute these air pollutants to specific sources. The current work deals with the design, construction and testing of a chamber for the generation of dust from samples matching exactly the emissions of a certain pollution source and the collection of PM10 particles on a PM10 sampler installed within. For the needs of our study, selected PM10 sources in the wider industrial area of Kozani-Ptolemaida were considered for sampling and the collected samples were analyzed by means of XRF.

Keywords: atmospheric pollution, receptor modeling, laboratory chamber, PM10 profile

1. INTRODUCTION
Urban, sub-urban and industrial areas face atmospheric pollution problems linked mainly to particulate matter (PM) emissions. In such areas, there can be found a great variety of emission sources, such as vehicles, industrial stacks, power plants etc, the emission rates of which ranging over several orders of magnitude. Since PM has harmful effects on human health and vegetation (Blanchard, 1999; Cesari et al, 2012) it is of great importance to identify the pollution sources and to quantify the atmospheric particulate matter. Considering the variety of sources and emissions in the aforementioned areas, together with the fact that PM can be transported in an area from distant sources, this may be difficult.

There is a variety of methods globally applied in order to link the air pollution sources to the measured pollution concentrations, named a) emission analysis, b) trend analysis, c)
tracer studies, d) trajectory analysis, e) receptor modeling and f) dispersion modeling. The reliability of the applied method affects directly and decisively the effectiveness of the subsequently adopted measures towards the air quality management.

Emission analysis is based on the fact that an accounting of emission rates by source type and location largely characterizes source contributions to ambient concentrations. In trend analysis, a comparison between the emission changes with changes in ambient concentrations or deposition fluxes of pollutants potentially provides information on source – receptor relationships. Tracer studies are of use for quantifying dispersion characteristics of plumes, providing empirical data for evaluating long-range trajectory models, characterizing atmospheric transport, and conducting material balances for use in quantitative source apportionment. Trajectory analysis is routinely used to estimate atmospheric transport for source attribution. Receptor models use the ambient pollutant measurements in the receptor to infer the source types and contributions that lead to pollutant concentrations at the receptor. Dispersion models input pollutant emission rates and meteorological information into a mathematical model that disperses (and may also chemically transform) the emitted pollutant, generating a prediction of the resulting pollutant concentration at a point in space and time (Blanchard, 1999).

From the aforementioned methods, receptor and dispersion modeling are mainly applied in order to link the air pollution sources to measured PM concentrations. The receptor - based source apportionment is considered as a top-down methodology while the dispersion – based one, as a bottom- up methodology (Guttikunda, 2009). The use of dispersion modeling (bottom – up approach) results in a) physical and quantitative identification of the potential sources in the studied region, b) description of the physical and chemical processes with potential to impact the advection and chemistry of the pollutants including the local topography and meteorological features, c) documentation of the formation of the secondary pollutants along with the primary ones, d) a baseline mapping of the pollution and hot spots for the area of interest and e) apportionment of the pollution by source. On the other hand, the top - down approach (receptor modeling) results in a) identification of hot spots with critical pollutant levels in the region, b) identification of the chemical composition of the particulate pollution, following the chemical analysis of the measured samples, c) description of the source impact estimates, d) differentiation of the primary and secondary pollution at the hot spots and e) apportionment of the pollution by source (Guttikunda, 2009).

The most widespread method for source apportionment is the receptor modelling approach, comprising of sampling in the study area by creating a network of proper instruments around the area, chemical analysis of samples and calculation of each source contribution to the air pollution of the area. The method provides information on the types of sources that are responsible for the observed pollutants, estimates of contribution of the sources for multiple locations over various time periods, average source strength at various hot spots and a basis to develop realistic and cost – effective strategies to reduce particulate pollution. However, it has the following disadvantages: a) it is not able to differentiate sources that have similar chemical composition, b) is not able to measure emission rates of individual sources, c) apportionments vary with meteorology and d) high cost related to monitoring of instruments and chemical analysis of samples (Guttikunda, 2009).

In order for any air quality management plan related to PM to be applied, it is of great importance to attribute these air pollutants to specific sources. Aiming to isolate each PM10 source, a dust generation chamber was designed, constructed and tested with samples matching exactly the emissions of a certain pollution source in an industrial area.
Within the chamber a PM10 sampler was installed while the collected samples were analysed by means of XRF.

2. DEVELOPMENT OF LABORATORY DUST – GENERATION CHAMBER

2.1. State-of-the-art
Laboratory dust generation chambers have been used for a variety for studies, from biomedical applications (Kaya et al, 1996) to wind erosion and fugitive dust research (Cowherd et al, 1992). The majority of these chambers are utilized in order to collect a physical aerosol sample on a filter medium for gravimetric analysis.

Early dust generators consisted of a “long, thin glass tube in which the time required for a given amount of materials to fall to the bottom was measured and related to particle’s sizes using Stoke’s Law”. Commercial and highly available off – the – shelf appliances such as blenders, laboratory flasks and agitators, rotating cylinders, jar mills with ceramic media, even commercial ventilation systems and air compressors, have been modified in order to generate dust from specific samples. In general, dust generators are classified according to the sample preparation and collection and to the method used for the generation of dust. In some chambers the dust is generated by the powders (particularly soils) in more or less the undisturbed, mixed state they exist in nature. On the other hand, there are systems in which the powder is sieved prior to being added to the chamber. Considering collection, some devices suspend a tiny amount of source material and attempt to collect as much of the dust emitted as possible, while others generate a large cloud of dust and the aerosol sampler collects only a small part of it (Gill et al, 2006).

The majority of dust production instruments can be divided in three classes according to the method used for aerosol generation: a) Fluidization (gas dispersion or ventilation), resulting in dust suspension by direct entrainment into airflow in a tube, b) Gravitation (drop method), in which the source sample falls as a discrete slug through the air into an enclosed chamber from which dust is evaporated and c) Mechanical dispersion or Agitation, involving the repeated fall of source material from top to bottom of a horizontal rotating cylinder or tube and its entrainment into air flow. The choice of any class of dust generation chambers is obviously related to the needs of the corresponding experiment and the specific property or process meant to be measured. A thorough review of the aforementioned dust generators can be found in literature (Gill et al, 2006).

Due to the importance of source apportionment studies, there is an on-going research related to the use of different resuspension chambers in order to generate fugitive geological source profiles, including the DRI resuspension sampling system (Chow et al, 1994), the UC Davis Resuspension Test Chamber (Carvacho et al, 2002), ventilated wooden chambers (Cesari et al, 2012) even rotating drums (Madden et al, 2010).

2.2. Design and Construction
The design of the dust generation chamber described in the current work was based on a method found in literature (ASTM, 1984). This method requires 200 g of material to be placed in the bottom of a “fluidizing bed dust chamber” consisting of a capped upright 7.62 cm diameter tube, 45.72 cm long with small holes in the top and bottom in which a line of regulated 50l min⁻¹ air flows, “puffing/pulling” the dust up into the chamber. The tube is to be clamped to a vibrator running at 29,000 vibrations min⁻¹, continuously agitated for 20 minutes and continuously sampled by an aerosol sampler. Considering the method of dust generator, it could be classified as a Fluidization chamber.

The whole installation, including the dust generation chamber and the PM10 sampler is presented in Figure 1(a). As shown in Figure 1, the dust generation chamber (Figure 2)
was adjusted to a closed sampling chamber (Figure 1d). For the needs of the construction of the dust generation chamber common laboratory glassware was used. The chamber consisted from i) one horizontal 5.6 cm diameter glass tube, 26 cm long, with three openings, one (right) capped with a Polyethylene (PE) cap, one (right) holding a Polytetrafluoroethylene (PTFE) filter in order to prevent PM10 particles coming into the chamber from the environment, and one in the middle from where it was connected by a flexible rubber tube to the ii) vertical 5.6 cm diameter glass tube, 56 cm long, having five capped side openings, one in the bottom and one in the top, and iii) a Polyvinyl chloride (PVC) tube, connected to the vertical glass tube by a flexible rubber tube, where the PM10 sampler was installed (Figure 1). The dust generation chamber was clamped on a laboratory vibrator (Figure 1) to facilitate the generation of dust.

**Figure 1.** (a) Dust generator and sampling installation. Embedded in the figure are also shown (b) PM10 sampler inlet, (c) filter holder and filter cassette and (d) sampling chamber

The materials for the whole installation design were chosen so as to minimise static electrical charges. The diameter and length of the PVC tube (Figure 1d) were chosen according to the corresponding dimensions of the PM10 sampler inlet (Figure 1b), so as to assure enough space for the dust to be suspended. The vertical glass tube was included in the whole installation unit for further experiments requiring the presence of gas flows.
2.3. **PM10 collection instrument**

A Low Volume Sampler LVS 3.1 provided by Derenda, was used for the collection of PM10. The device is used to collect particulates from outdoor air in compliance with corresponding standards (EN 12341, 1998; EN 14907, 2005). The air throughput volume when collecting PM10 or PM2.5 fractions amounts to 2.3 m$^3$ h$^{-1}$. The device is equipped with a 4 m$^3$ rotary vane vacuum pump. Maximum vacuum at the filter is 300 mbar, while the maximum air throughout when using a glass fibre is 3.5 m$^3$ h$^{-1}$. The sampling time can be adjusted from 5 minutes to 1000 hours.

The unit is installed in a stainless steel housing 30 cm in width, 45 cm in height and 25 cm in depth. The inlet is 20 cm long, has a diameter of 8.4 – 14 cm and is connected to the unit through a hollow stainless steel tube 23.3 cm long (Figure 1b). Temperature, relative humidity and pressure in the inlet are continuously measured. The diameter of the filter holder and the filter cassette is 55 and 41 mm, respectively (Figure 1c).

3. **TESTING OF CHAMBER**

3.1. **Description of sampling sites and strategy**

Soil samples corresponding to various activities inside an open pit mine in the wider Ptolemais – Kozani area were collected following the procedure described in literature (EPA 454-R-93-037, 1993). The samples included: unpaved road dust, fly ash, lignite, mixture of fly ash and overburden and low quality lignite ore. All samples were placed to sealed PE bags and delivered to the laboratory for preparation prior to dust generation.

3.2. **Sample preparation, dust generation and collection**

Filters were processed prior and after experiments according to literature (EN 12341, 1998). Before any use, PTFE filters were exposed for 48h on open but dust protected sieve-trays in an air-conditioned weighing room with a temperature of (20±1) °C and a relative humidity of (50±5) % before weighing, in order to remove any loose fibre material. The dust loaded filters were equilibrated under the same conditions before weighing.

The samples were dried in an oven for 24 h at 100°C. After drying the samples were sieved through a 200-mesh Tyler (75μm) screen (Kong et al, 2011; Samara 2005) to remove larger particles that do not contribute to dust and to PM10 emissions. About 10 g of sieved material was placed into the horizontal dust generation chamber (Figure 2) and different dust generation modes were tried: a) operation of vibrator, b) operation of sampler and c) simultaneous operation of vibrator and sampler. Modes a) and b) were
not sufficient to generate dust from neither sample therefore it was decided to simultaneously operate the vibrator and the sampler, the latter contributing to the dust generation.

### 3.3. Dust generation and sampling conditions

During the operation of the whole installation, temperature, relative humidity and pressure on the filters were simultaneously measured. The measurements are shown in Table 1.

<table>
<thead>
<tr>
<th>Sample #</th>
<th>Material</th>
<th>Temperature orifice (°C)</th>
<th>Relative Humidity (%)</th>
<th>Filter pressure (mbar)</th>
<th>Air volume (m³)</th>
<th>Air throughput volume (m³/h)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Low quality lignite ore</td>
<td>22,50</td>
<td>32,00</td>
<td>25,20</td>
<td>0,15</td>
<td>2,25</td>
</tr>
<tr>
<td>2</td>
<td>Low quality lignite ore</td>
<td>22,50</td>
<td>33,70</td>
<td>24,10</td>
<td>0,38</td>
<td>2,28</td>
</tr>
<tr>
<td>3</td>
<td>Low quality lignite ore</td>
<td>21,70</td>
<td>33,50</td>
<td>25,50</td>
<td>0,57</td>
<td>2,29</td>
</tr>
<tr>
<td>4</td>
<td>Lignite ore</td>
<td>15,80</td>
<td>33,50</td>
<td>23,70</td>
<td>1,14</td>
<td>2,29</td>
</tr>
<tr>
<td>5</td>
<td>Fly ash</td>
<td>18,50</td>
<td>20,30</td>
<td>41,40</td>
<td>0,57</td>
<td>2,29</td>
</tr>
<tr>
<td>6</td>
<td>Fly ash</td>
<td>19,00</td>
<td>20,40</td>
<td>27,50</td>
<td>0,19</td>
<td>2,26</td>
</tr>
<tr>
<td>7</td>
<td>Fly ash and overburden mixture</td>
<td>22,90</td>
<td>33,20</td>
<td>24,30</td>
<td>0,19</td>
<td>2,26</td>
</tr>
<tr>
<td>8</td>
<td>Fly ash and overburden mixture</td>
<td>21,90</td>
<td>33,50</td>
<td>31,50</td>
<td>0,57</td>
<td>2,29</td>
</tr>
<tr>
<td>9</td>
<td>Unpaved road dust</td>
<td>19,20</td>
<td>35,40</td>
<td>43,10</td>
<td>0,57</td>
<td>2,29</td>
</tr>
<tr>
<td>10</td>
<td>Unpaved road dust</td>
<td>20,10</td>
<td>35,10</td>
<td>31,60</td>
<td>0,38</td>
<td>2,28</td>
</tr>
<tr>
<td>11</td>
<td>Unpaved road dust</td>
<td>20,70</td>
<td>34,90</td>
<td>32,90</td>
<td>0,19</td>
<td>2,26</td>
</tr>
</tbody>
</table>

As shown in Table 1, the air throughput volume was close to the instrument’s specifications (2.3 m³/h), while filter pressure was a factor of PM10 concentration on the respective filter. Air volume changed according to the duration of each experiment (Table 2). Temperature and relative humidity were continuously measured in the sampling chamber.

Operation time of both vibrator and sampler was a factor of the sample, as also presented in Table 2. Since the collected PM10 samples are to be analysed by XRF, the mass of the filter deposit was measured after each experiment and presented in the same Table 2.

In samples 4 - 11 shown in Table 2, vibrator started to operate 1 min prior to sampler operation. During the experiments, it was found out that, due to the design of the chamber, the operation of the vibrator alone resulted in the collection of the sample in the left side of the horizontal chamber, near to the filter intake (Figure 2). Upon the sampler operation, the sample was close to the air flow therefore a higher amount of dust could be generated.

According to literature (EPA - 625/R-96/010a, 1999) the minimum mass filter loading required for multi-elemental XRF analysis is about 15 μg/cm² and the maximum 400 μg/cm², with an optimum value at about 100 μg/cm². Considering this, the required time for dust generation chamber could be estimated for each material studied, from the corresponding PM10 concentration. As shown in Table 2, the time required for the collection of the appropriate mass from a low quality lignite sample was 5 minutes simultaneous operation of vibrator and sampler. In the case of lignite ore, 15 minutes simultaneous operation of vibrator and sampler, with 1 minute additional operation of vibrator prior to sampler operation, resulted in the collection of only 20 μg/cm² PM10, a concentration close to the lower PM10 concentration limit for XRF (15 μg/cm²). For the fly ash (samples # 5 and 6), the mixture of fly ash and overburden (samples # 7 and 8) and the sample from unpaved road (samples # 9-11), 5 minutes of simultaneous operation
with 1 minute additional operation of vibrator prior to sampler operation was adequate for the collection of the appropriate PM10 mass (Table 2).

### Table 2. Dust generation mode (simultaneous operation of vibrator and sampler)

<table>
<thead>
<tr>
<th>sample #</th>
<th>material</th>
<th>vibrator operation (min)</th>
<th>sampler operation (min)</th>
<th>PM10 (μg/cm²)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>low quality lignite ore</td>
<td>5</td>
<td>5</td>
<td>119</td>
</tr>
<tr>
<td>2</td>
<td>low quality lignite ore</td>
<td>10</td>
<td>10</td>
<td>212</td>
</tr>
<tr>
<td>3</td>
<td>low quality lignite ore</td>
<td>15</td>
<td>15</td>
<td>268</td>
</tr>
<tr>
<td>4</td>
<td>lignite ore</td>
<td>16 plus 16</td>
<td>15 plus 15</td>
<td>20</td>
</tr>
<tr>
<td>5</td>
<td>fly ash</td>
<td>16</td>
<td>15</td>
<td>4319</td>
</tr>
<tr>
<td>6</td>
<td>fly ash</td>
<td>6</td>
<td>5</td>
<td>214</td>
</tr>
<tr>
<td>7</td>
<td>fly ash and overburden mixture</td>
<td>6</td>
<td>5</td>
<td>190</td>
</tr>
<tr>
<td>8</td>
<td>fly ash and overburden mixture</td>
<td>16</td>
<td>15</td>
<td>660</td>
</tr>
<tr>
<td>9</td>
<td>unpaved road dust</td>
<td>16</td>
<td>15</td>
<td>1488</td>
</tr>
<tr>
<td>10</td>
<td>unpaved road dust</td>
<td>11</td>
<td>10</td>
<td>360</td>
</tr>
<tr>
<td>11</td>
<td>unpaved road dust</td>
<td>6</td>
<td>5</td>
<td>230</td>
</tr>
</tbody>
</table>

### 4. CONCLUSIONS

The creation of a pollution sources inventory and the subsequent quantification of each source contribution to the measured pollutants concentrations is the first step in addressing the atmospheric pollution problem faced by industrial and urban areas. Among the various methods for source attribution, receptor modelling is the most widespread one, as it provides information on the types of sources that are responsible for the observed pollutants, estimates of contribution of the sources for multiple locations over various time periods, average source strength at various hot spots and a basis to develop realistic and cost-effective strategies to reduce particulate pollution. However, it comprises some drawbacks, such as difficulty in differentiating sources that have similar chemical composition, inability to measure emission rates of individual sources, strong dependence and variability of apportionments with meteorology, and high cost.

In order to overcome the aforementioned disadvantages, a laboratory dust generation fluidization chamber was designed and constructed from ordinary laboratory glassware and materials minimizing static electrical charges. The chamber was clamped on a laboratory vibrator and adjusted to a closed sampling chamber where a PM10 sampler was placed. Initial experiments, using dried and sieved samples matching exactly emissions of various PM10 sources from an open pit mine, showed that dust was able to be generated by the simultaneous operation of both vibrator and sampler.

The experiments also revealed the operation time required for the collection of the proper PM10 concentration for XRF analysis. One minute operation of vibrator prior to PM10 sampler operation resulted in a higher amount of dust generated. The subsequent five minutes simultaneous operation of both the vibrator and PM10 sampler was adequate for the sampling of the required for XRF analysis PM10 concentration, for the majority of the samples. Exception was the lignite ore sample, for which, after 30 minutes of simultaneous operation, the PM10 concentration collected was close to the lower concentration limit for XRF analysis.

Future work includes testing of more samples from the region under study and multi-elemental XRF analysis of all the collected PM10 samples. Experiments will be also carried out introducing various gases, while the chamber will be modified in order to be able to collect PM10 from exhaustions from combustion engines.
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REFERENCES