A ROUTINE PROCEDURE FOR THE MEASUREMENT OF PARTICLES OR FIBRES RELEASED FROM CIGARETTES OR FILTERS

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Summary

The release of particles or fibres from cigarettes during smoking has been the subject of much debate in recent years. A number of studies quantify the number and type of particles or fibres released from cigarettes or filters during puffing. Many of these methods involve dry puffing followed by relatively complex procedures to identify the number and nature of the particles or fibres released. A more rapid procedure to estimate the number of particles released is required to allow screening of a wide range of products. A procedure using laser particle counters, used to monitor air quality in a range of applications and offer a rapid measurement of total particle quantity, is recommended.

A routine method for particle counting is described based on a modified laser particle counter and flow switching system. This method allows particles eluted from cigarettes or filters in the size range from 0.5 to 50 microns and at a range of different flow rates to be rapidly counted.

To demonstrate the effectiveness of the procedure using laser particle counters, typical data are then given for the particle release of carbon manufactured from a range of raw materials for use in cigarette filters, such as coconut, coal, wood and peat.

Background

The topic of fibre and particle release from cigarettes has been comprehensively studied over the last twenty years. The main conclusions of this work was that for current commercial products the number of fibres released are low and too large to be respirable[1] and that any particles released are also low in number especially when compared to the normal particle count in ambient air and do not pose a risk to the smoker[2].

Over the last twenty years the methods used for measuring particle and fibre release have increased in complexity. Early methods included a simple tap test[3] where the end of a filter or cigarette was pressed against a sticky surface such as agar gel and the fibres or particles trapped in the gel were counted optically. Product was also dry puffed (puffs drawn using a smoking machine without lighting) using a smoking machine and any released particles or fibres trapped using a microporous membrane and counted using optical microscopy[3, 4]. More sophisticated analytical techniques have also been used to identify the type of fibres or particles released including Scanning Electron Microscopy, Energy Dispersive X-Ray and Infrared Spectrometry[4]. Other workers have used Raman Spectroscopy[5]. The size of fibres released has also been measured for dry puffing and during smoking using inertial impactors[1, 6].

More recently dry puffing has been used in combination with laser particle counters[2, 5] for the estimation of particle and fibres released from cigarettes and filters. Laser particle counters offer a much simpler and rapid method of estimating the release of particles and fibres from cigarettes and filters.

Laser particle counters are used for a variety of applications in the measurement of air quality. These include clean room monitoring, pollution monitoring and process control. Most commercial instruments operate by drawing air through the instrument at a constant flow rate. As a particle passes through a laser the light is scattered or blocked. This light blocking or scattering is detected and a voltage pulse generated corresponding to the size of the particle. Generally, the commercial instruments measure between 6 to 8 size ranges and the data are sorted to give a count according to the appropriate size range. Particle sizes as low as 0.3 μm and as high as 500 μm can be measured, however, multiple instruments would be required to cover the full range.

As previously stated one application of laser particle counters is in air monitoring. In most environments the number of small particles is quite large. Typically, for rural outdoor air in the UK, the particle count in a volume of 500 ml would be over 1000 particles smaller than 2.5 μm, and over 10 particles in the 2.5 to 10 μm size range. Particles larger than 10 μm would be much less abundant: generally less than 1 in 500 ml of air. Out door air particle counts next to major roads or in city centres would be much higher. Indoor air quality differs greatly according to the location but can be higher or lower than the out door air. For example, in Filtrona's Scientific Services laboratory, established for the testing of cigarettes and filters, the typical particle count in 500 ml of air is 200 particles smaller than 10 μm in size.

The high background count means that apparatus used for particle counting in cigarettes and filters must be located in a clean environment as the numbers of particles and fibres eluting from a cigarette or filter are much lower than typical background levels.

A Routine Procedure for the Measurement of Particles or Fibres Released from Cigarettes or Filters

A schematic diagram of an apparatus to measure the fibre and particle release is shown in fig 1 below. The main elements are a clean cabinet, ISO class 4, which is used to house the samples under test and provide a low background count, typically approaching zero. An airflow meter is used to set the flow rate to the required value usually 1.05 L/min (the average flow for a 35 ml puff over a two second period is equivalent to 1.05 L/min) or higher for more intense testing. The air flow meter is only used to set flow and was not in the flow stream during the actual testing.

After particles have been eluted during constant flow they are passed directly in to the laser particle counter. The instrument used here was a Lasair II particle counter that divides the counted particles in to 6 size ranges, namely, 0.5 to 1, 1.0 to 5.0, 5.0 to 10.0, 10.0 to 25.0, 25.0 to 50.0 and greater than 50 μm.

For all measurements, samples must be handled carefully to avoid dust pick up or stressing the sample in any way that could create dust. For any testing twenty replicate determinations are carried out. The laser and clean cabinets are switched on and allowed to stabilise and the airflow set to the required value usually 1.05 L/min. Each sample is then carefully inserted in to the measuring head so that the mouth end is towards the laser counter. Air is drawn through the sample for 30 seconds and the number of particles eluted counted by the laser. At the end of the measurement the particle count is displayed on the front panel and can also be automatically printed by the instrument.

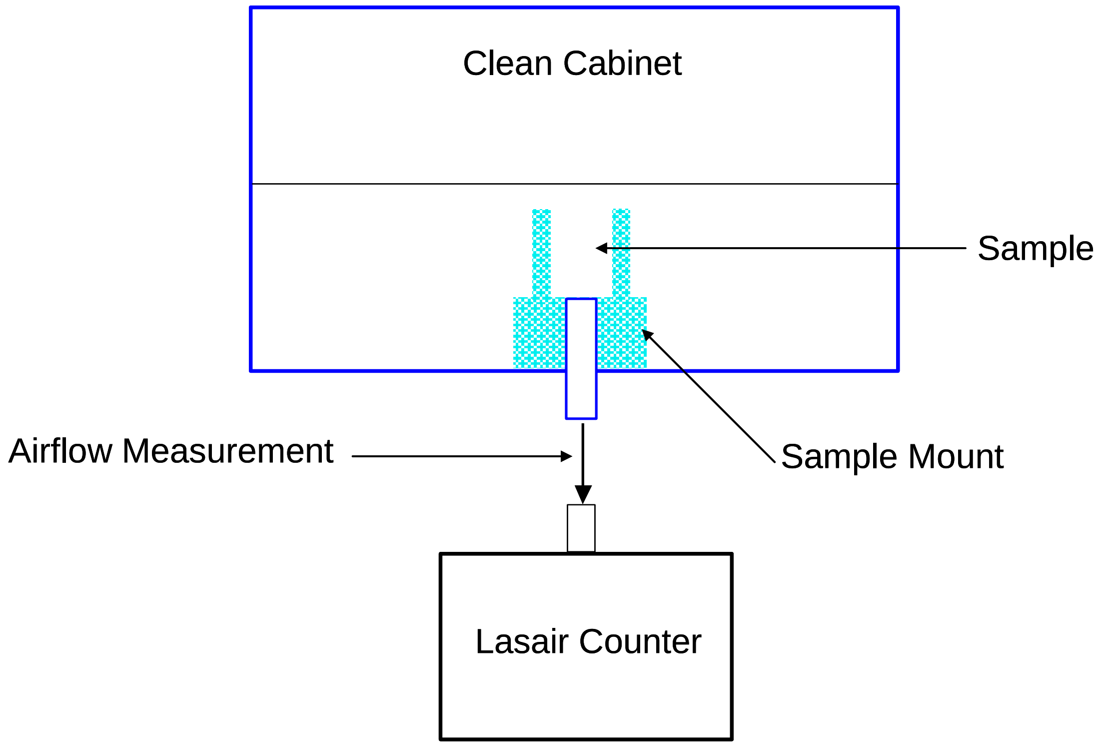

Figure 1 Schematic Diagram of Particle Counting Apparatus

Demonstration of a Routine Procedure for the Measurement of Particles or Fibres Released from Filters or Cigarettes

A study has been carried out to compare the particle count given by a number of filters, especially to compare the effect of carbons made from different feedstocks on particle counts.

All of the filters tested were 27 mm long with a circumference of 24.4 mm. The filter pressure drop was approximately 70 mm and the tar retention approximately 50 %. Filters containing carbon were produced as active acetate dual filters with a 12 mm long acetate mouth end segment and a 15 mm carbon on tow segment. All the carbon filters contained 60 mg of carbon per filter.

Five different carbon types were studied and the properties of these carbons are shown in table 1 below. These were the standard carbon made from coconut shell - normally used in cigarette filters - and carbons produced from coal, wood, peat and a synthetic spherical carbon. The activity values given in the table are the equilibrium adsorption of cyclohexane vapour.

Carbon Type	Particle Size mm	Moisture %	Bulk Density g/ml	Activity %	pH
Coconut	0.21 – 0.60	14.7	0.57	30	9.7
Coal	0.21 – 0.60	6.9	0.57	36	8.1
Wood	0.21 – 0.60	11.2	0.32	29	9.5
Peat	0.21 – 0.60	13.2	0.40	37	9.3
Synthetic	0.30 – 0.40	3.4	0.58	48	8.8

Table 1 Carbon Properties

While the vast majority of carbon used in cigarette filters are made from coconut shells, coal carbon and to a lesser extent the synthetic spherical carbon has also been used in commercial products. Figure 2 below gives a comparison of the particle release for these three carbons compared to the acetate filter using the routine procedure described above.

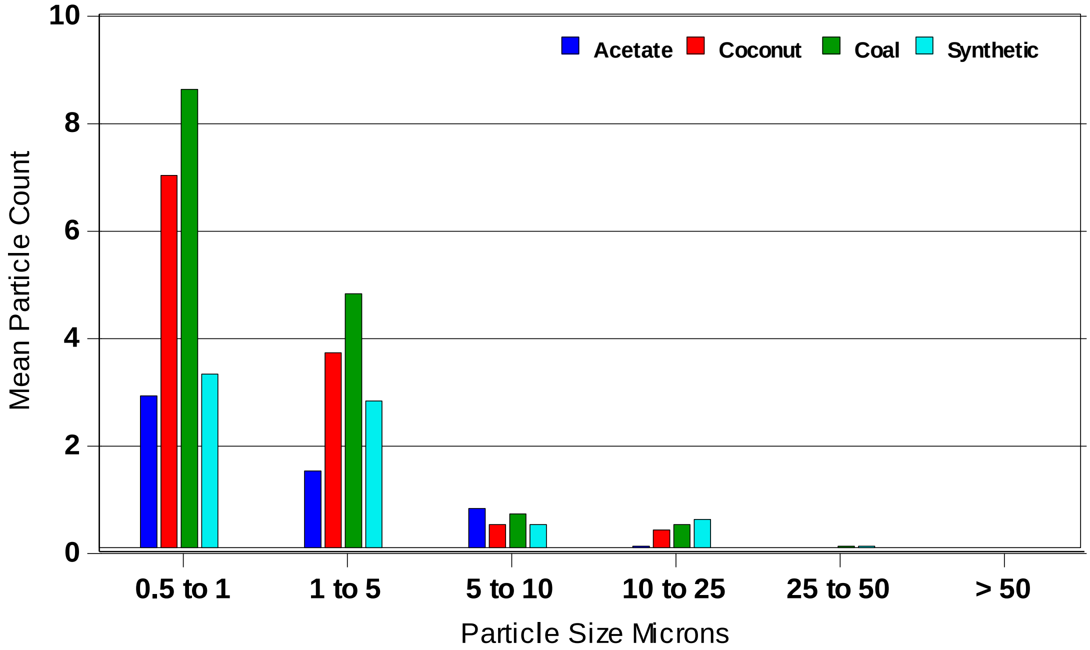

Figure 2 Mean Particle Count

The data show that for all the filters, as would probably be expected, the smaller particles are more readily eluted in to the air stream. In general the number of particles is quite small with the total mean number of particles released being acetate 5.3, synthetic carbon 7.3, coconut carbon 11.6 and coal carbon 14.7. For all of the measurements taken using the laser equipment detection of particles above 50 μm in size was very rare.

For other carbon types, however, much larger particle counts are found. If the data for the wood and peat carbons are added to the above plot significantly higher particle counts are seen for these carbons. These data are shown in figure 3 below.

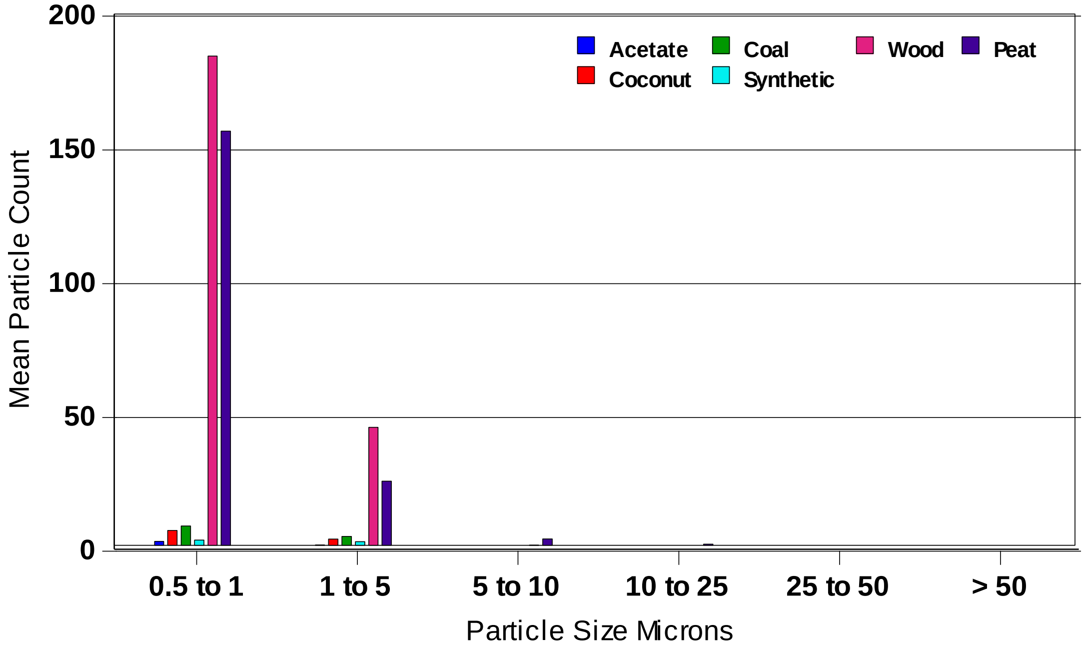

Figure 3 Mean particle Count All Samples

Again the number of particles in the smaller size range 0.5 to 1.0 μm is much higher than the larger particles. For these carbons, however, the total particle counts are 232.0 for the wood carbon and 187.5 for the peat carbon significantly higher for any of the other carbons.

The data demonstrate that the method is able to differentiate between carbons from different feedstocks in terms of particle release.

This procedure is also applicable to test cigarettes. For cigarettes typical particle counts are generally slightly higher than for filters, for example, a Kentucky reference cigarette (K3R4F) had a measured particle count of 8.6 particles per cigarette with a similar particle size range distribution to that found for filters. The measured values per cigarette were size range 0.5 to 1.0 μm; 4.4 particles, size range 1.0 to 5.0 μm; 3.4 particles, size range 5.0 to 10.0 μm; 0.6 particles and above 10 μm; 0.2 particles.

All of the preceding data has been derived at a flow rate of 1.05 L/min and higher flow rates would be equivalent to more intense smoking. It may be expected that flow rate would have an effect on the number and perhaps size of particles released from cigarettes or filters. The flow rate of 1.05 L/min for standard testing was based on a 35 ml puff taken over a two second duration. The effect of flow rate has been tested over a range of 4 flows: 0.75, 1.05, 1.65 and 2.25 L/min, which are equivalent to puff volumes of 25, 35, 55 and 75 ml over a two second duration.

Two filters have been tested on a K3R4F cigarette: a monoacetate filter and a carbon dual filter (27 mm long with a 12 mm acetate segment and 15 mm carbon segment containing 60 mg of coconut shell carbon). A plot is shown in figure 4 below
demonstrating the relationship between total particles released and flow rate for both samples tested.

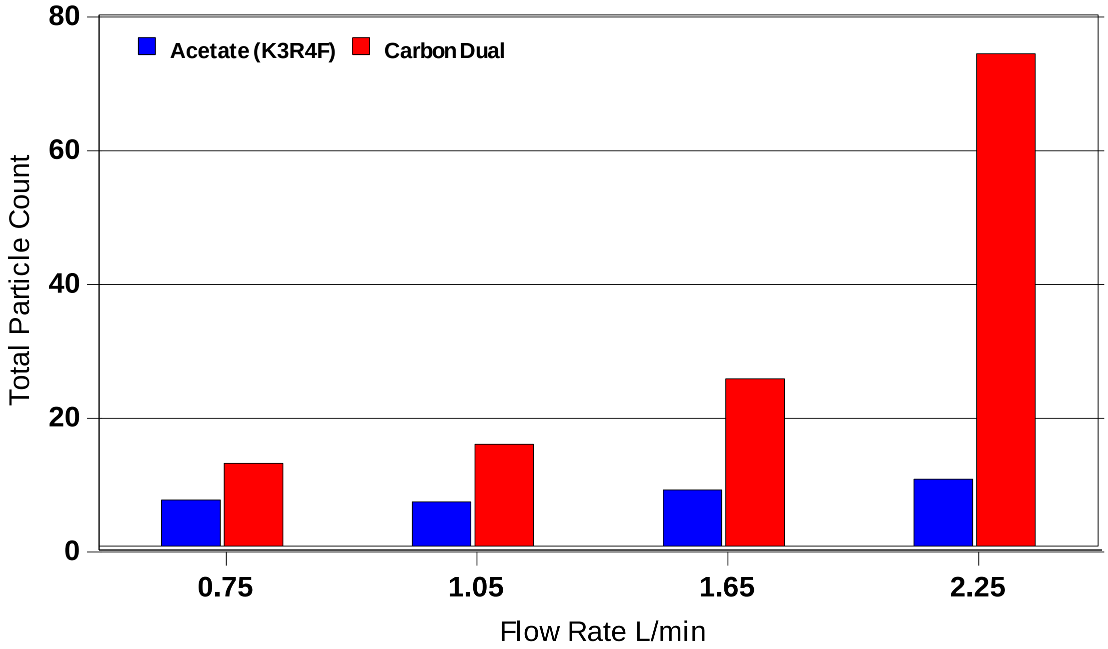

Figure 4 Effect of Flow rate on Total Particles Released

As perhaps would be expected, as the flow rate increases, the number of particles released increase. The effect is small for acetate filters and the increase in value is within the variation of the method. In this series of measurements the total number of released particles increased from 7.2 at a flow of 1.05 L/min to 8.9 at a flow of 1.65 L/min and 10.6 at a flow rate of 2.25 L/min.

For carbon dual filters, however, the increase is much more pronounced. The number of particles released for an equivalent 55 ml puff (1.65 L/min) approaches twice the number released for an equivalent 35 ml puff (1.05 L/min).

A more detailed examination of the carbon dual filter in figure 5 below gives the relationship between flow rate and particle release showing the particle count in each size range.

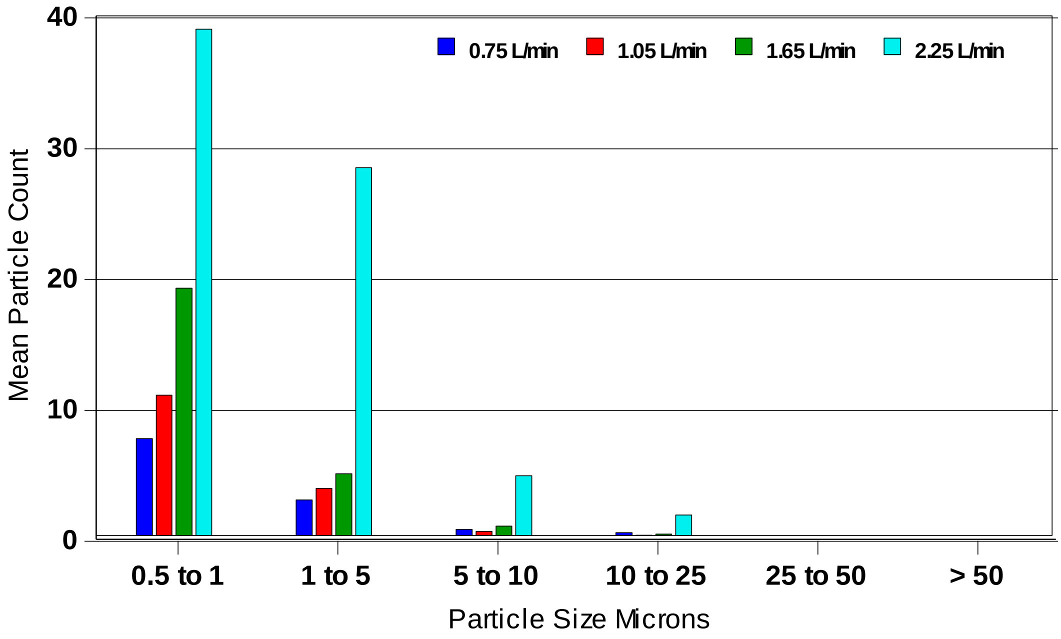

Figure 5 Flow Rate and Particle Release for a Carbon Filter

It can be seen that the largest increase in particles are given for the smaller particles mainly smaller than 5 μm. For the very small particles in the < 1 μm size range the number released approximately doubles as the flow rate increases from 1.05 to 1.65 L/min, and then doubles again when the flow rate increases to 2.25 L/min.

For larger particles an increase in number of particles released is only given for the highest flow rate. As the flow rate increases small increases in particle counts are seen, but for particles between 1 to 25 μm the particle counts at 2.25 L/min are approximately 5 times that found for the 1.65 L/min flow rate. At the highest flow rate, about one particle above 25 μm is found for every 20 filters tested.

The procedure using laser particle counters can demonstrate the changes in particle release across higher flow rates and therefore more intensive smoking regimes.

Discussion

A rapid low cost method has been developed for the determination of fibres and particles released from cigarettes and filters. This simple procedure shows that the number of particles released from current commercial products is much lower than those found in ambient air and can differentiate between different sources of carbon in terms of particle release. The use of a procedure with laser particle counters also allows studies to be made at different flow rates to represent different intensities of puffing.

It does not, however, allow the particles released to be identified and cannot be used for testing of a lit product. Samples have to be handled carefully before measurement because any contamination from dust can increase the apparent number of particles released. Measured values are usually quite variable and coefficients of variation for 20 replicate determinations of 50 % or higher are not unusual.

Nonetheless, this routine procedure is recommended to provide the rapid measurement of particles released. It fulfils the need for efficient screening for a wide range of products. Furthermore, by creating a substantial dataset this procedure could be used to establish a measurable quality standard for fibre and particle release for all products across the industry.

References

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