We would like to thank the reviewers for their positive and constructive reviews of this paper. We address the specific points of each reviewer below. Reviewer comments in blue, author response in black, text added or amended in paper in purple.

Review 1

In this paper, Stewart et al. present emission factors (EFs) for non-methane volatile organic compounds (NMVOC) produced during residential solid fuel combustion for heating and cooking in Delhi, India. Common cooking fuels were collected from across New Delhi and burned under controlled laboratory conditions. NMVOC emissions were measured using multiple gas chromatography-based systems and a proton transfer-reaction time-of-flight mass spectrometer. Species-specific and total measured NMVOC EFs are reported in the paper/supplement for each fuel type burned and the results are discussed in the context of similar laboratory studies of residential biomass burning emissions. The authors find that for most fuels oxygenated NMVOCs account for the largest proportion of the total NMVOC emissions. Additionally, they report that the speciation and total measured NMVOC emissions vary widely between different fuel types, highlighting the need for a more complete understanding of residential biomass burning EFs. The study focuses on fuels that are specific to India and New Delhi, making their results relevant to local and regional chemical models. Emission factors from this study are also more broadly applicable to other regions where residential solid fuel combustion is used as the primary means of cooking and heating. This clearly written manuscript addresses an underrepresented area of biomass burning emissions with a very comprehensive NMVOC EF dataset. I particularly appreciated the inclusion of LPG emissions to demonstrate its potential as a ‘cleaner’ alternative. I recommend this paper for publication after addressing the minor issues discussed below.

1) My main concern is the representativeness of the burning chamber used in this study to the common stoves used in residential settings. Although the chamber description is referenced, I feel that it is important for this study to include a more detailed description of the stove/combustion chamber itself along with how it was operated to replicate real-world conditions as the combustion efficiency is well known to influence NMVOC EFs.

The chamber was based on a previously published design of Venkataraman and Rao, (2001). The chamber was designed to simulate the convective nature of biomass combustion, so it was important to ensure that the processes studied here of emissions entrainment into the hood were also convection driven so that they did not exert a draft which altered combustion conditions. The dilution setup employed here was optimised to give dilution ratios of 40-60, which allowed cooling of gases to around 2-3 °C above ambient temperature at the top of the flue.

This chamber has been previously tested and optimised to ensure that conditions replicate those of a natural draft during combustion. The burn rate has been previously evaluated using extraction rates of 0.01-0.03 $m^3 s^{-1}$ and stove-hood distances of 0.35-0.65 m. Larger extraction rates and stove-hood distances less than 0.45 m enhanced burn rates above the natural burn rate. Stove-hood distances above 0.65 m resulted in emissions not being captured by the hood. The optimum conditions were used of 0.45 m between sample and hood with a flow rate of ~ 0.022 $m^3 s^{-1}$.

We now include additional details of the chamber used in the main text and a detailed schematic of the chamber is given in the supplementary information. We also include a video abstract which shows the ignition of a sample during this study to show the conditions this study was designed to replicate. Whilst several different stove types can be used in India, this study was most like a traditional fire. The main text has now been changed.

Fuels were burnt at the CSIR-National Physical Laboratory (NPL), New Delhi, under controlled conditions utilizing a combustion chamber based on the design of Venkataraman and Rao, (2001). Several previous studies have been based on this chamber design (Venkataraman and Rao, 2001; Venkataraman et al., 2002; Saud et al., 2011; Saud et al., 2012; Singh et al., 2013), which was designed to simulate the convection-driven conditions of real-world combustion and is displayed in the Supplementary Information S1. The burn-cycle used in this study was adapted from the VITA water-boiling test, which is designed to simulate emissions from cooking, using expert local judgement to ensure conditions replicated real-world burning conditions. The cycle included emissions from both low- and high-temperature burning conditions, as these are encountered in real cooking practice and should give a more reflective NMVOC emission factor.

Fuel (200 g) was placed 45 cm from the top of the hood and rapidly heated to spontaneous ignition, with emissions convectively driven into a hood and up a flue to allow enough dilution, cooling and residence time to achieve the quenching typically observed in indoor environments. These conditions have been previously optimised to ensure that emissions entrainment into the hood did not exert a draft which altered combustion conditions.

This schematic of the burning chamber has been added to the supplementary information.

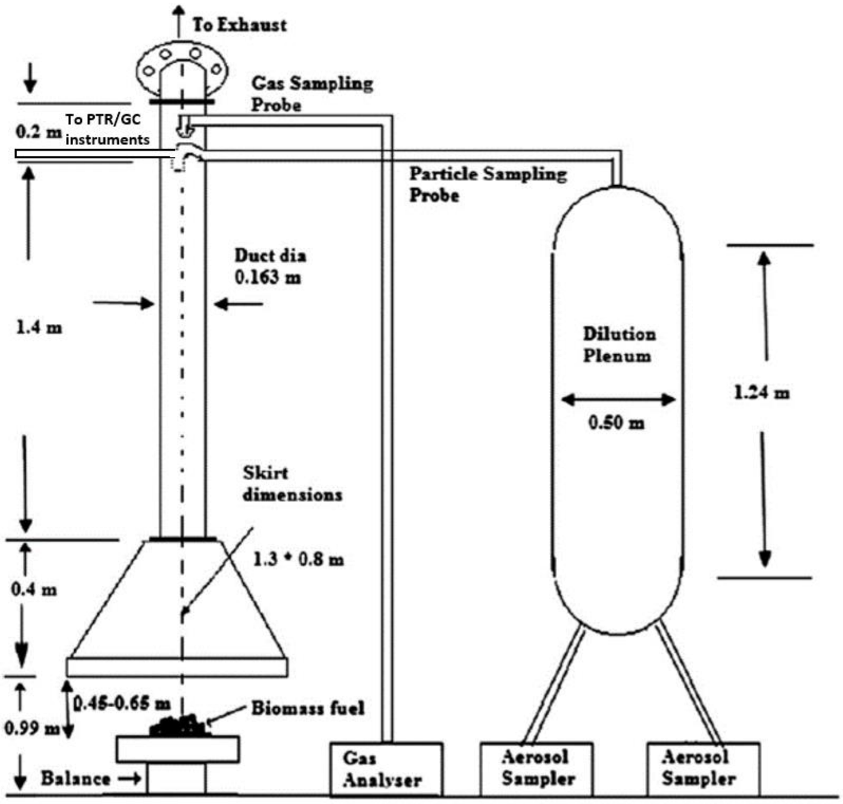

Figure S1. Schematic of combustion chamber used for experiments.

2) There is very little discussion about the error associated with the reported EFs beyond that associated with each instrument, nor is the EF variability between repeated burning experiments of similar fuels included. For example, the mean EFs in the supplement and Table 2 should be associated with the fire-to-fire variance, such as the standard deviation of the burns.

The standard deviation of measured NMVOC emission factors by sample type are presented in Table 1 in the main text. We also now look at the EF variability of similar fuels in detail within the main text, as detailed below.

Figure 8 shows the distribution of total measured NMVOC emission factors for fuel wood, cow dung cake, crop residues and MSW. Boxplots show the mean, median, interquartile range and range within 1.5IQR. The solid circles display the spread of measured emission factors by fuel type. The zoomed green region given in Figure 8B specifically focuses on the variability in emission factors of individual species of fuel wood, which has been explored in detail due to the large number of samples. Repeat samples collected from the same location are shaded in grey. For fuel wood, measured NMVOC emission factors varied by over a factor of 20 between 4.3-96.7 g kg^{-1}. The NMVOC emission factors showed a right skewed distribution with a median of 11.7 g kg^{-1}, mean of 18.7 g kg^{-1} and an interquartile range of 15.3 g kg^{-1}. For repeat measurements of identical species of fuel wood collected at the same location, except for *Ficus religiosa*, measured emission factors from repeat experiments varied over a much smaller range, by up to a factor of 2.3. Variation between emissions from these samples were likely due to different moisture contents of actual samples measured and the specific combustion conditions of individual burns. Despite the samples for *Holopetlea spp* and *Eucalyptus spp* coming from different locations, emission factors for these samples were quite reproducible and only varied by a factor of 1.2-1.5. For remaining identical species of fuel wood collected from different locations, emission factors varied over a much larger range by factors of ~ 2-9.

For the crop residue species studied here, NMVOC emissions were right skewed with a with a median of 29.5 g kg^{-1} which was less than the mean of 37.9 g kg^{-1} and varied from 8.9-73.8 g kg^{-1} with an interquartile range of 53.9 g kg^{-1}. *Cocos nucifera* and *Solanum melongena* were repeat measurements of fuel collected from the same location and varied by factors of 1.8-2. NMVOC emissions from *Brassica spp* fuel, which was collected from different locations, varied by a factor of ~ 8. Cow dung cake and MSW samples were all collected from different locations and varied by up to factors of up to 2.4 and 2.1, respectively.

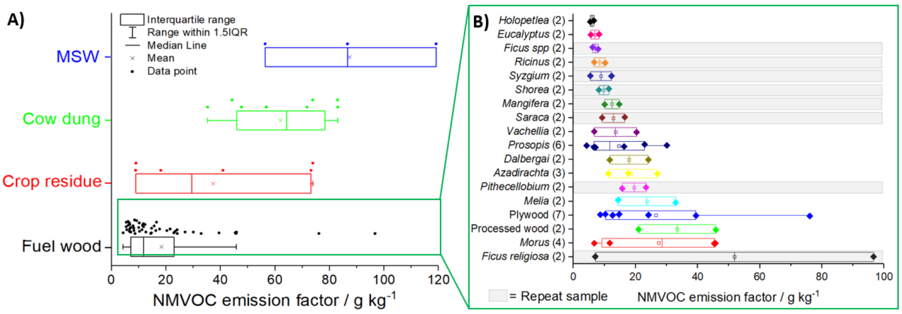

Figure 8. Variability in NMVOC emission factor by fuel type. A) = Range of emission factors measured for fuel wood, cow dung cake, crop residue and municipal solid waste samples with box plots showing the mean, median, interquartile range, range within 1.5IQR and solid circles showing the spread of measured emission factors by fuel type. B) = Zoomed green region displaying range of NMVOC emission factors measured for individual species of fuel wood with grey shaded region indicating repeat samples from the same sample collection location and diamonds indicating the measured NMVOC emission factors.

Similarly, what is the error associated with the stack flow-based method for determining EFs?

Venkataraman and Rao, (2001) studied the stack-flow based method for determining EFs. As part of this study the reproducibility of dilutions from repeat fires was examined, with Table S1 giving the results at 1 σ of 4 repeat measurements.

Table S1. Repeatability of dilution ratios using stack flow-based method, taken from Venkataraman and Rao, (2001).

Sample	Dilution ratio
Wood	57 ± 6
	47 ± 7
	46 ± 8
	53 ± 3
Biofuel briquette	40 ± 3
	42 ± 7
Dung cake	56 ± 13
	42 ± 9
	43 ± 6
	60 ± 7

3) It is unclear whether CO and CO_2 were measured during the experiment, but if available their inclusion as EFs and MCEs for each burn would greatly help anchor this study in the context of NMVOC emission literature as the authors discuss in the conclusion.

CO and CO_2 were originally intended to be measured during this study, however, due to a technical failure there was a lack of sufficiently reliable data to be of use to include here. The authors acknowledge the importance of emission factors to CO and CO_2 as well as the influence of modified combustion efficiency to NMVOC emissions from burning studies, but unfortunately cannot include this here.

Additional comments by line:

110 – Add missing word: due ‘to’ vehicular emissions.

This is now corrected.

198 – Repeated word ‘given’. 414 – Should this be referencing S4?

This is now corrected.

S2 appears to show how EFs were calculated. Additionally, S2, describing how EFs were calculated, is not referenced in the text and should be added. This also goes for the tables in supplement S4, they should be referenced in the text and would benefit from having the individual tabs labeled (Sx).

The text has been changed to read, where S2 is now S3 due to reordering.

Figure 7 shows a detailed breakdown of the mean NMVOC emission factors by fuel type measured for all 76 burns (see the Supplementary Information S3 for values). Emission factors have been determined by calculating the mean NMVOC concentrations up the flue over a 30-minute period, in line with the GC sample time, with any small emissions after this sample window not included. This has been related to the total volume of air convectively drawn up the flue and the mass of fuel burnt (see the Supplementary Information S4 for details).

The tabs in the supplementary table are now labelled too.

498 – Discussion of total emission factors would be more accurately discussed as total 'measured' emission factors as the techniques used in this study likely miss a portion of low volatility species, which could be lost in sample inlets and chromatography columns.

We now use the terminology total measured emission factor throughout.

502-508 – It is unclear if you included the GC measurements in your total emission factors from this discussion. Is that the purpose of discussing proton affinities here? If so just state that alkanes and alkenes measured by the GC's were included in the total EF as appears to be described in S3.

This is now stated.

509 – Should this be S3? Or maybe relevant to both S3 and S4 'EF g kg' tab? 512 – Include the mass of benzaldehyde.

This is now clarified in the text.

544 – Stockwell et al. (2015), which the authors compare results to, define IVOC/SVOC as species with a molecular weight greater than toluene. Is there a reason the authors instead choose to define IVOCs as those with molecular weight greater benzaldehyde?

This is a good question, as saturation vapour concentration pressures depend on both mass and functionality and it is therefore difficult to define based on a particular mass. IVOCs are defined as having effective saturation concentration, C^* =300-3×10^6 µg m^{-3}. We calculated these for NMVOCs in our mass spectra following the approach given in Lu et al, (2018).

The estimated C^* for toluene was ~ 1.4×10^8 µg m^{-3} and benzaldehyde ~ 7×10^6 µg m^{-3}. For this reason, we based the IVOC boundary on benzaldehyde and not toluene. The total amount of IVOCs presented from this study would therefore be a more conservative estimate of total measured IVOC compared to Stockwell et al. (2016). The reason for this approach has been emphasised in the text, and attention also brought to this approach being approximate. The text has been changed to read:

IVOCs are defined as having effective saturation concentration, C^*, =300-3×10^6 µg m^{-3} (Donahue et al., 2012). The C^* of several species was estimated using a previously established approach (Lu et al., 2018), with the IVOC boundary defined in this study at benzaldehyde (*m*

= 106.12) for which C^* was ~ 7×10^6 µg m^{-3}. Table 2 also shows an approximation for the mean amount of IVOCs released by fuel type. This approach was approximate as vapour pressures depend on both mass and functionality. The fuels tested in this study showed that mean emissions of IVOC species represented approximately 18 – 27% of total measured emissions from all fuel types other than LPG. This demonstrated that biomass burning is potentially a large global source of IVOCs. Further studies are required to better understand the contribution of IVOC emissions from biomass burning to SOA formation.

587 – Figure 9A – The different studies in the plot are very difficult to distinguish and it is unclear what the authors are trying to convey with it due to the potential comparison of unrelated EFs. For example, what does the inclusion of 'all species reported in review and comparable studies' include? Are wood emission factors from this study compared to garbage burning or peat EFs for Stockwell et al. (2016)? Are EFs from western US wildfires (Liu et al., 2017) and south eastern US agricultural fires (Muller et al., 2016) relevant to this work? Further, EFs also vary between fuels due to differences in combustion efficiency (e.x. dung, peat, and trash will smoulder more than wood) whereas this figure implies fuel type is the only difference. It would make sense to have this as a more direct comparison between related fuels (i.e. just literature fuel wood, cow dung, etc.).

This is a good point. We made the comparison to just woods from Akagi et al. (2011), Andreae (2019) and Koss et al (2018). Generally, the emission factors measured were larger from these studies than measured in our study. We also compared emission factors from waste burning in Stockwell et al. (2016) to our study. Despite this, the comparison was not particularly interesting. When we plotted these different studies, the other studies often had higher emission factors than our study for many data points, but there was considerable scatter in the data points both above and below the 1:1 line. This meant that including these additional plots in the main body of the text was not particularly beneficial, so we removed this section from the paper.

650-657 – As mentioned, if available, this study would also greatly benefit from reporting CO, CO2, and MCE values for each burn. As the authors state, this would allow their EFs to be evaluated based on the impacts of MCE. Additionally, reporting MCE would allow these results to be more accurately compared to other studies, while CO and CO2 are themselves important inputs for climate models.

This is covered as part of the response to major comment 3.

Review 2

In this work, the authors measured emission factors of volatile organic compounds (VOCs) emitted from combustion of a variety of fuels commonly used in India. This is an important topic for both atmospheric chemistry and human health, since domestic fuel combustion is associated with one of the leading causes of morbidity and mortality globally. The authors used a comprehensive suite of analytical techniques, and measured emission factors for a wide array of VOCs. In particular, the combination of PTR-MS and multiple GC techniques is highly complementary and provides detailed information about the emissions. In addition, the fuels studied are very commonly used in India and would provide important data and insights. From experimental design to data analysis and interpretation, this work is of the highest quality and potential impact.

We thank the reviewer for such a positive review and for highlighting the strengths of this work.

I recommend publication in ACP, and my suggestions and comments here are minor and for reference only. One of the major strengths of this study is its direct relevance. The authors stated that they used “expert local judgement to ensure conditions replicated real world burning conditions”. It is unclear what that means from a technical standpoint. Precisely what variables are replicated to reflect local practices? (e.g. fuel types, forms of fuel, humidity etc.) How should future studies replicate the results presented here?

This has been partly covered by the response to main comment 1 from reviewer comment 1 through the additional discussion on chamber setup and design. Fuels were collected from residents of Delhi state from the same areas they collected their fuels to be burnt. This was designed so that the fuels which were burnt were identical to those burnt under real circumstances. The fuels were handled and stored as locals would to ensure that the moisture content of samples was like those being burnt for residential energy requirements. The combustion chamber used has been previously studied to ensure the combustion conditions were convective and that neither the hood nor any fans nor pumps within the setup created a draft which altered combustion conditions and in turn NMVOC emissions.

Similar studies would follow the experimental design given in Venkataraman and Rao, (2001), which is now also given in the Supplementary Information. The fuel samples were collected from a detailed study, which in summary split the state of Delhi into 66 5×5 km grids and conducted fuel usage surveys at almost 700 locations of over 6000 households. The collected fuels were designed to reflect the results of this survey. The results of this survey will be presented in a separate publication (Mondal et al., 2021) led by the Indian co-authors in this study.

In multiple instances, the authors noted that PTR-MS measured higher amounts than GC techniques for the same compounds, and attributed to “unidentified isomers”. I am curious to learn more about this issue. If a particular PTR-MS m/z is assigned to a compound that has multiple isomers, and in GC there is an associated peak (which represents one of the isomers), shouldn’t the comparison be made between PTR-MS m/z and the sum of all isomers measured by GC (i.e. sum of multiple peaks)? If the “unidentified isomers” are not observed by the GC, that would imply these “unidentified isomers” are chemically different from the proposed compound, and therefore PTR-MS is actually misidentifying these isomers.

This is indeed one of the limitations of measurements with PTR-ToF-MS. Figure 6 gives a comparison of some aromatic compounds measured by the PTR-ToF-MS and both GC instruments. The PTR-ToF-MS instrument gives a signal at a particular mass, for which we have assigned the most probable identity. If we take the example of benzene, other potential C_6H_6 compounds include benzvalene, bicyclopropenyl, fulvene, prismane, 3-Methylidenepent-1-en-4-yne, Hexadiyne, 1,3-Hexadiyne, 1,4-Hexadiyne, 1,5-Hexadiyne, 2,4-Hexadiyne, Hexadienyne, 1,2-Hexadien-4-yne, 1,2-Hexadien-5-yne, 1,3-Hexadien-5-yne, 1,5-Hexadien-3-yne or 2,3-Hexadien-5-yne. All these other C_6H_6 compounds would be indistinguishable from benzene.

The GC instruments only have flame ionisation detectors, and so we are only able to calibrate peaks for which the identity is known through the retention time of a known standard compound. Peaks for other C_6H_6 compounds may be present in our chromatograms, but we are not able to identify which these are. As a result, this comparison is currently benzene on the GC×GC-FID, benzene and coeluting peaks with the same retention time on the DC-GC-FID and all C_6H_6 compounds on the PTR-ToF-MS. The purpose of this comparison was therefore to show that the instruments were all measuring similar concentrations, but there may be some additional undistinguishable structural isomers measured on the PTR-ToF-MS instrument.

As a further complication, NMVOCs on the PTR-ToF-MS were calibrated with the rate constant for the reaction of the hydronium ion with the NMVOC of the most likely compound. Where multiple isomers were present, this may lead to slight mis quantification.

It is not surprising that cow dung cake and municipal solid waste had the highest emission factor, but what is the typical quantity burned? I imagine the fuel wood would be much more commonly used. It might be useful to clarify whether with the high emission factors of cow dung cake and municipal solid waste translate to higher contributions of VOCs.

This is a great comment and something we have prepared a further study on which is currently awaiting submission. It is also difficult to put a firm number on, as there are considerable uncertainties in fuel consumption estimates for India and many estimates are quite out of date. We have added the following text to help clarify this.

Considerable uncertainties exist in consumption estimates for fuels such as cow dung cake and municipal solid waste in India. A previous study estimated that in 1985 in India fuel wood consumption was 220 Tg and cow dung cake consumption 93 Tg (Yevich and Logan, 2003). A different study made an India-wide estimate for 2000 which estimated fuel wood consumption to be 281 (192-409) Tg and cow dung cake consumption to be 62 (35-128) Tg (Habib et al., 2004). A more recent study estimated fuel wood usage at 256 Tg and cow dung cake consumption at 106 Tg for 2007 (Singh et al., 2013). Estimates of the amount of municipal solid waste burnt in India are even fewer than for cow dung cake consumption. Two previous studies have estimated that 81.4 Tg of municipal solid waste was burnt in India in 2010 (Wiedinmyer et al., 2014) and that 68 (45-105) Tg was burnt in 2015 (Sharma et al., 2019). The mean emission factors for cow dung cake and municipal solid waste combustion were considerably larger than for fuel wood and highlight that at an India-wide level these may represent a significant NMVOC source.

Figure 3 shows an interesting trend: there seems to be two rather distinct phases of burning in A and B. Was this typical of all burns? If so, why is that the case, and what do these phases represent?

The phases may represent flaming and smouldering emissions. Sekimoto et al. (2018) showed that during lower temperature pyrolysis there were greater emissions of heavier molecular weight phenolic and furanic components. It may also be due to the off gassing of IVOC species from the quartz filter or the tubing used in this laboratory setup, which becomes more pronounced when the concentration of other gases is lower once the burning experiment has almost finished.

We are cautious to offer too much explanation of the difference in phases of the burn because the method used to measure total gas flow up the flue was used to give an integrated picture of the whole burn, and not used for a time-resolved measurement. Whilst this gives a quantitative measurement of the total volume of air sampled up the flue over the entire burn, it may slightly misrepresent the phases in Figure 3 where the average gas velocity over the entire burn has been used to calculate the concentration. Certain regions of the burn may be more (or less) pronounced here, which are not accounted for with this approach.

Minor comments: Line 37: “400 Tg yrˆ-1” and “annually” in line 38 are redundant

This has now been corrected.

Line 110: due to Line 131: unclear what 6000-7000 kt yrˆ-1 is referring to. Is it total VOCs?

This is a reference to total VOC emissions from burning in India. The text has been changed to read

India-specific inventories which include residential burning indicate a considerable emission source of total NMVOCs of around 6000-7000 kt yr^{-1}

Line 162: range of Line 181: does the quartz filter potentially remove gas phase species such as IVOCs?

This is a good comment, and it may and is therefore one of the limitations of this study. Between each sample there was a considerable amount of particulate matter collected onto these filters and if we did not change the filter between samples it increased the background on the PTR-ToF-MS instrument. This may indicate some off gassing of species which may have partitioned to the aerosol phase. We would not want to run these instruments without a filter to remove particulate matter but feel that this is an important limitation, so we now acknowledge it in the text later with the discussion section about the proportion of IVOC species. It may also explain the larger mass fraction of IVOC species in region C of Figure 3 as this could be species off gassing from chamber walls or this filter, which enhances the proportion of IVOC species.

In addition, this may represent an underestimate because the quartz filter placed on the sample line may remove IVOC species which have partitioned to the aerosol phase due to the high aerosol concentrations present during source testing.

Figure 1: I understand that the x-axis is referring to longitude, but I initially mistook it as ion.

Thank you for highlighting this, we have now capitalised these axes so that they are clearer.

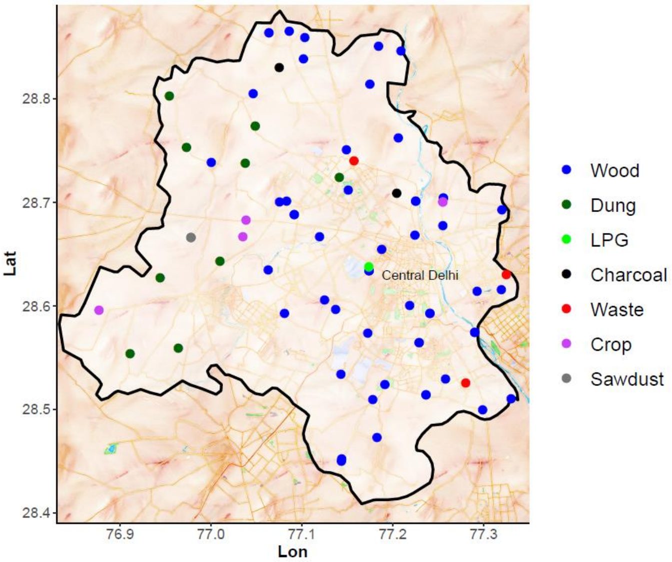

Figure 1. Locations across New Delhi used for the local surveys into fuel use and collection of representative biomass fuels. Map tiles by Stamen Design. Data by © OpenStreetMap contributors 2020. Distributed under a Creative Commons BY-SA License.

Line 306: Since this is the results section, the title "Chromatography" is not very helpful. I suggest a more descriptive title.

Thank you for this suggestion, we have changed this to read "Comparison of chromatograms obtained from combustion of different fuel types".

Figure 2: Are the units of the color scale arbitrary? Are each of the samples obtained with the same volume of air sampled? If so, it might be useful to clarify and emphasize that, because if the color scales and the air volume sampled were the same, then solid waste and cow dung are indeed emitting more VOCs.

Thank you for highlighting this. We have chosen this contrast scale between 0 and 25 as it allows a nice visualisation and comparison of the NMVOCs present. All the chromatograms are at the same level of contrast and samples were collected with the same sample volumes. We added to the caption on figure 2 Samples A-D were collected with the same sample collection parameters and the chromatograms are set at the same contrast level to allow direct comparison between different fuel types.

Figure 3: it is not directly obvious to me that from Region A to C the average m/z is increasing. Perhaps show the median mass, or overlay these diagrams, or stack them vertically with a common x-axis?

Thank you for this suggestion, we have now stacked the binned mass spectra of regions A and B vertically to better emphasise this change. We have removed the section from 500-700 seconds at this was showing a similar trend as 200-300 seconds.

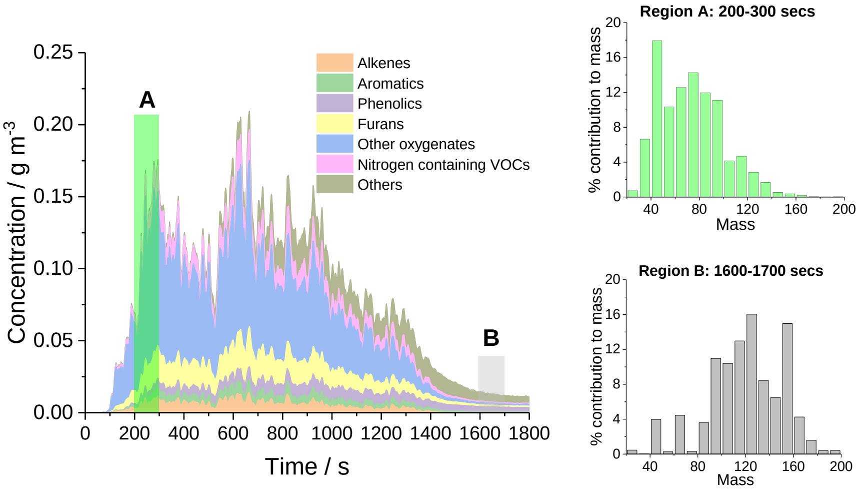

Figure 3. PTR-ToF-MS concentration-time series during the first 30 minutes of a cow dung cake burn coloured by functionality with regions A and B displaying mass spectra placed into *m*/*z* bins of 10 Th. Fuel collected from Pitam Pura, New Delhi.

Line 380: How is ACES an abbreviation of broadband cavity-enhanced spectroscopy?

Thank you for pointing this out, we have changed it to read airborne cavity-enhanced spectroscopy (ACES).

Line 383: 1 +/- 30% can be misleading. I suggest 1+/- 0.3 Line 387:

Thank you for the suggestion, we have changed this to read 1 ± 0.3%.

“These previous comparisons underline the challenges faced with quantitative NMVOC measurements. . .” this sentence seems to contradict the previous sentences. It seems that correlation coefficients are generally >0.8 in the literature, which is the same as what was obtained in this study. It seems to be this level of consistency is to be expected. (Perhaps that’s what the authors mean?)

Thank you for this suggestion, we have changed this section to read These previous comparisons indicate the level of consistency expected with instrument comparisons of quantitative NMVOC measurements from burning experiments.

Figure 7 may be too detailed and many of the labels are far too small to see. I struggle to see the message conveyed by these figures. I suggest showing figures that support the discussion in 3.4 and minimize information overload.

Thank you for this comment, we agree that this figure is too small. This was partly due to the need for portrait figures in ACPD. We have changed it so that it covers 2 full pages in landscape format. We have also added some additional shaded boxes to help highlight which areas correspond to specific fuel types.

We would like to keep the information presented, to allow readers to quickly glance by class of NMVOC to see if it is important to their study or interest. We feel presenting this graphically is easier for users to determine if classes of VOC are important for particular fuel types than looking through the table in the supplementary information which contains 76 rows of different burns and almost 200 columns of NMVOCs.

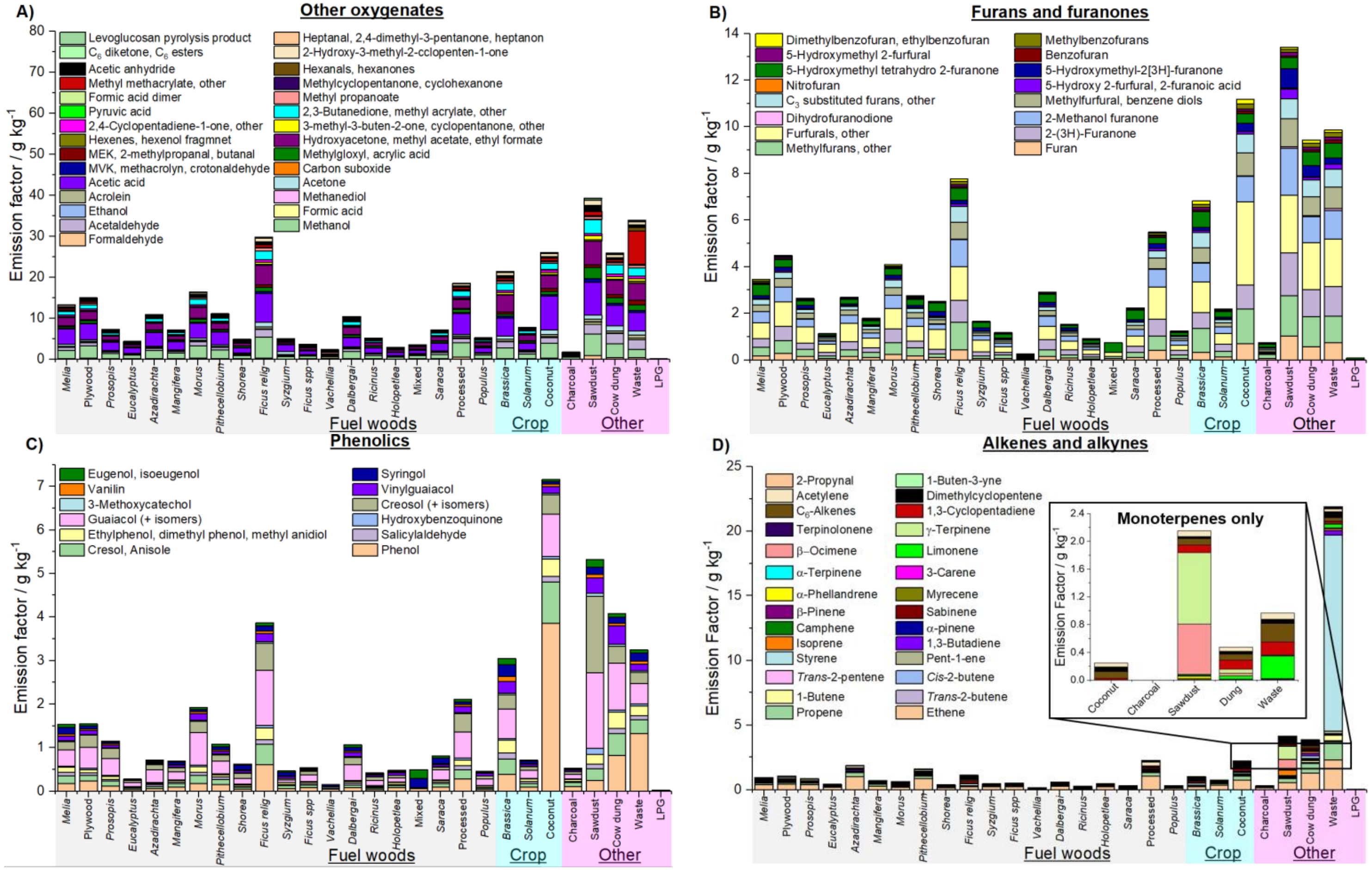

Figure 7. Measured emission factors grouped by functionality.

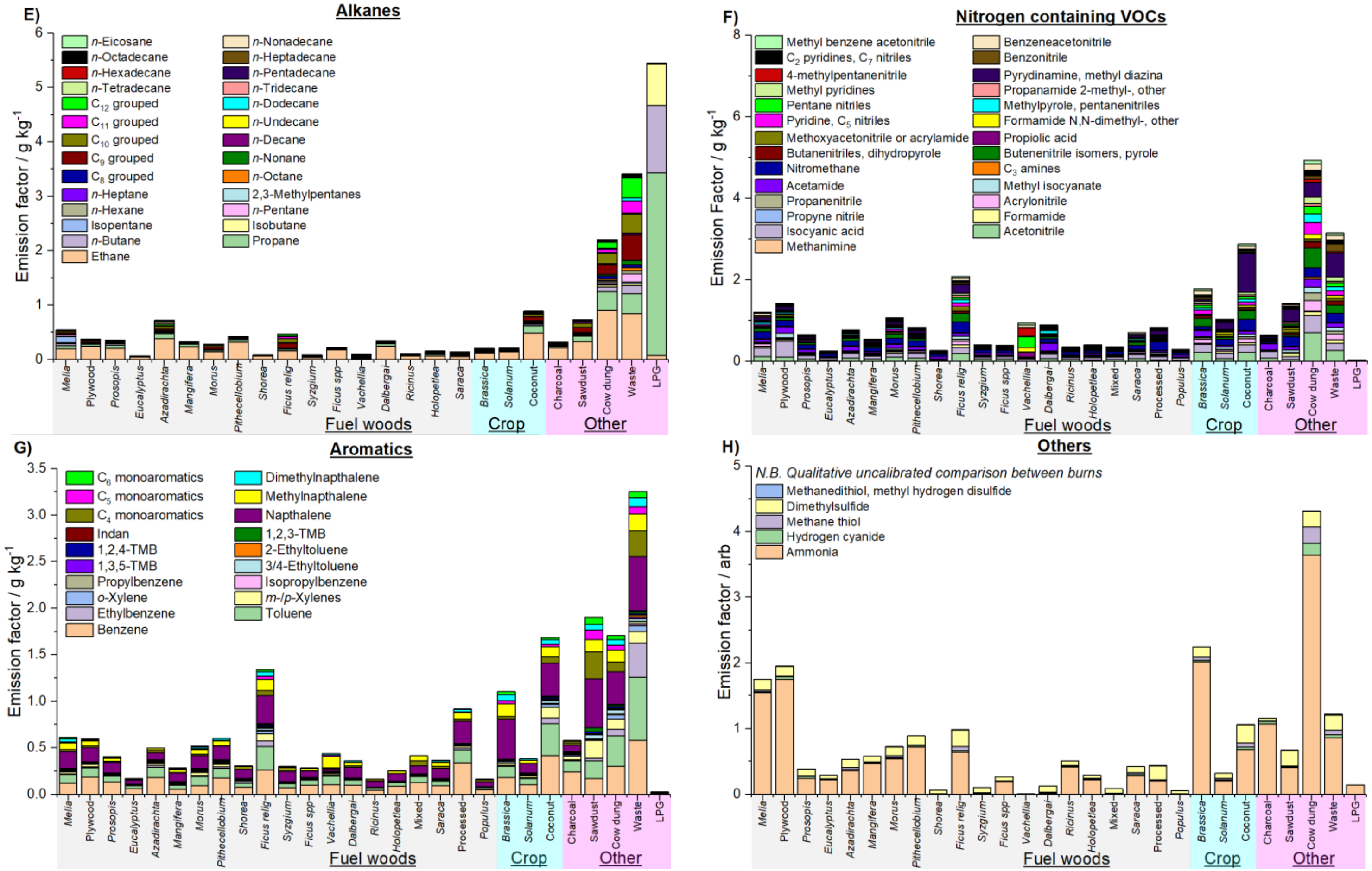

Figure 7 continued.

Line 528: “however” might not be the best conjunction. “But” is more grammatically correct.

Thank you for this suggestion, we have changed this to but.

Line 547: Since IVOCs are being reported, what is the typical PM concentration? Are the PM concentrations high enough for IVOC to partition into the particle phase?

Thank you for this great question. Traditional source studies make separate gas-phase and particle-phase measurements of organic emissions, and so if the study is only of gas-phase emissions and IVOCs partition to the particle phase because of the unrealistically high particulate matter concentrations during source testing then these are not accounted for and therefore underestimated in the emission factor measurement. The gas-phase emission factors presented in this study may therefore represent an underestimate. We now acknowledge this in the text with the previous comment about discussion of the quartz filter.

We attempt to help overcome this as part of a further study, which is currently in review with a different journal, where we map the emissions from the DC-GC-FID, GC×GC-FID, PTR-ToF-MS and SPE/PTFE-GC×GC-ToF-MS analyses onto a volatility basis dataset to evaluate organic emissions across the entire volatility range and remove this traditional gas/aerosol phase divide when analysing sources at the point of emission.

Emissions of non-methane volatile organic compounds from combustion of domestic fuels in Delhi, India

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Abstract

29 different fuel types used in residential dwellings in northern India were collected from across ~~New~~ Delhi (76 samples in total). Emission factors of a wide range of non-methane volatile organic compounds (NMVOCs) (192 compounds in total) were measured during controlled burning experiments using dual-channel gas chromatography with flame ionisation detection (DC-GD-FID), two-dimensional gas chromatography (GC×GC-FID), proton-transfer-reaction time-of-flight mass spectrometry (PTR-ToF-MS) and solid-phase extraction two-dimensional gas chromatography with time-of-flight mass spectrometry (SPE-GC×GC-ToF-MS). ~~94% quantification~~94% speciation of total measured NMVOC emissions was achieved on average across all fuel types. The largest contributors to emissions from most fuel types were small non-aromatic oxygenated species, phenolics and furanics. The emission factors (in g kg^{-1}) for total gas-phase NMVOCs were~~:~~ fuel wood (18.7, 4.3-96.7), cow dung cake (62.0, 35.3-83.0), crop residue (37.9, 8.9-73.8), charcoal (5.4, 2.4-7.9), sawdust (72.4, 28.6-115.5), municipal solid waste (87.3, 56.6-119.1) and ~~liquified~~liquefied petroleum gas (5.7, 1.9-9.8).

The emission factors measured in this study allow for better characterisation, evaluation and understanding of the air quality impacts of residential solid fuel combustion in India.

1. Introduction

Biomass burning is the second largest source of trace gases to the troposphere, releasing around a half of global CO, ~ 20% of NO and ~ 8% of CO_2 emissions (Olivier et al., 2005; Wiedinmyer et al., 2011; Andreae, 2019). ~~Biomass burning releases an estimated 400 Tg yr^{-1} of non-methane volatile organic compounds (NMVOCs) annually (Akagi et al., 2011)~~Biomass burning releases an estimated 62 Tg yr^{-1} of non-methane volatile organic compounds (NMVOCs) (Andreae, 2019), and is the dominant source of both black carbon (BC) and primary organic aerosol (POA), representing 59% and 85% of global emissions respectively (Bond et al., 2013). Biomass burning includes open vegetation fires in forests, savannahs, agricultural burning and peatlands (Chen et al., 2017) as well as the biofuels used by approximately 3 billion people to meet their daily cooking and heating energy requirements worldwide (World Bank, 2017). A wide range of trace gases are released from biomass burning, in different amounts depending on the fuel type and the combustion conditions, meaning that detailed studies at the point of emission are required to accurately characterise emissions. The gases released lead to soil-nutrient redistribution (Ponette-Gonzalez et al., 2016; N'Dri et al., 2019), can themselves be toxic (Naeher et al., 2007) and can significantly degrade local, regional and global air quality through the photochemical formation of secondary pollutants such as ozone (O_3) (Pfister et al., 2008; Jaffe and Wigder, 2012) and secondary organic aerosol (SOA) (Alvarado et al., 2015; Kroll and Seinfeld, 2008)~~ as well as~~. They can also lead to indoor air quality issues~~.~~ (Fullerton et al., 2008).

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Emissions from biomass burning and their spatial distribution remain uncertain and estimates by satellite retrieval vary by over a factor of three (Andreae, 2019). Bottom-up approaches use information about emission factors and fuel usage. However, information for many developing countries, where solid fuel is a primary energy source, is particularly sparse. Toxic pollution from burning has been linked to chronic bronchitis (Akhtar et al., 2007; Moran-Mendoza et al., 2008), chronic obstructive pulmonary disease (Dennis et al., 1996; Orozco-Levi et al., 2006; Rinne et al., 2006; Ramirez-Venegas et al., 2006; Liu et al., 2007; PerezPadilla et al., 1996), lung cancer (Liu et al., 1993; Ko et al., 1997), childhood pneumonia (Smith et al., 2011), acute lower respiratory infections (Bautista et al., 2009; Mishra, 2003) and low birth weight of children (Boy et al., 2002; Yucra et al., 2011). Smoke from inefficient combustion of domestic solid fuels is the leading cause of conjunctivitis in developing countries (West et al., 2013).

The harmful emissions from burning also resulted in an estimated 2.8-3.9 million premature deaths due to household air pollution (Kodros et al., 2018; World Health Organisation, 2018; Smith et al., 2014), of which 27% originated from pneumonia, 18% from strokes, 27% from ischaemic heart disease, 20% from chronic obstructive pulmonary disease and 8% from lung cancer, with hazardous indoor air pollution responsible for 45% of pneumonia deaths in children less than 5 years old (World Health Organisation, 2018)~~.~~(World Health Organisation, 2018). For this reason, hazardous indoor air pollution from the combustion of solid fuels has been calculated to be the most important risk factor for the burden of disease in South Asia from a range of 67 environmental and lifestyle risks (Lim et al., 2012; Smith et al., 2014).

The emissions from biomass burning fires are complex and can contain many hundreds to thousands of chemical species (Crutzen et al., 1979; McDonald et al., 2000; Hays et al., 2002; Hatch et al., 2018; Stewart et al., 2020a). Measurements of emissions by gas chromatography (GC) have been made (EPA, 2000; Wang et al., 2014; Gilman et al., 2015; Stockwell et al., 2016; Fleming et al., 2018), as it has the potential to provide isomeric speciation of emissions. However, it is of limited use in untargeted measurements from burning due to the complexity of emissions, leading to large amounts of NMVOCs released not being observed. Some of the main issues are that GC does not provide high time resolution measurements and several instruments with different column configurations and detectors are required to provide information on different chemical classes. Samples can also be collected into canisters or sample bags and then analysed off-line (Wang et al., 2014; Sirithian et al., 2018; Barabad et al., 2018), which can increase time resolution, but can also lead to artefacts (Lerner et al., 2017).

Recent developments have allowed the application of proton-transfer-reaction mass spectrometry (PTR-MS) to study the emissions from biomass burning (Warneke et al., 2011; Yokelson et al., 2013; Brilli et al., 2014; Stockwell et al., 2015; Bruns et al., 2016; Koss et al., 2018). PTR-MS uses proton transfer from the hydronium ion (H_3O^+) to ionise and simultaneously detect most polar and unsaturated NMVOCs including aromatics, oxygenated aromatics, alkenes, furanics and nitrogen containing volatile organic compounds ~~(NVOCs)~~ in gas samples. PTR-MS can measure at fast acquisition rates of up to 10 Hz over a mass range of 10 – 500 Th with very low detection limits of tens to hundreds of pptv (Yuan et al., 2016). The more recently-developed technique of proton-transfer-reaction time-of-flight mass spectrometry (PTR-ToF-MS) has allowed around 90% of total measured NMVOC emissions in terms of mixing ratio from burning experiments to be ~~quantified~~speciated (Koss et al., 2018) and has also been used to study the formation of SOA (Bruns et al., 2016). The main disadvantages of the PTR-ToF-MS technique are its inability to speciate isomers~~/isobars~~, significant fragmentation of parent ions, only being able to detect species with a proton affinity greater than water and the formation of water clusters needing to be ~~taken into account~~ considered (Stockwell et al., 2015; Yuan et al., 2017). More recently, measurements have also been made using iodide chemical ionization time-of-flight mass spectrometry (I^--CIMS), which is well suited to measuring acids and multifunctional oxygenates (Lee et al., 2014) as well as isocyanates, amides and organo-nitrate species released from biomass burning (Priestley et al., 2018). Multiple measurement techniques used in concert are therefore complementary, with the use of PTR-ToF-MS and simultaneous gas chromatography often alleviating some of the difficulties highlighted above.

Since the start of the century, rapid growth has resulted in India becoming the second largest contributor to NMVOC emissions in Asia (Kurokawa et al., 2013; Kurokawa and Ohara, 2019). However, effective understanding of the relative strength of different sources and subsequent mitigation has been limited by a deficiency of suitably detailed, spatially disaggregated emission inventories (Garaga et al., 2018). ~~Current~~A current receptor-model ~~studies have~~study has shown elevated NMVOC concentrations at an urban ~~sites~~site in Delhi to be predominantly due to vehicular emissions, with a smaller contribution from solid fuel combustion (Stewart et al., 2020b). However, approximately 60% of total NMVOC emissions from India in 2010 were ~~shown~~estimated to be due to solid fuel combustion (Sharma et al., 2015). Other studies have also suggested that burning may lead to enhanced concentrations of pollutants such as polycyclic aromatic hydrocarbons in Delhi (Elzein et al., 2020). A need has therefore been identified to measure local source profiles to allow evaluation with activity data to better understand the impact of unaccounted and unregulated local sources (Pant and Harrison, 2012).

Approximately 25% of worldwide residential solid fuel use takes place in India (World Bank, 2017, 2020), with approximately 25% of ambient particulate matter in South Asia attributed to cooking emissions (Chafe et al., 2014). Despite large government schemes, traditional solid fuel cookstoves remain popular in India because they are cheaper than ones that use ~~liquified~~liquefied petroleum gas (LPG) and the meals cooked on them are perceived to be tastier (Mukhopadhyay et al., 2012). The total number of biofuel users has been sustained by an increasing population, despite the percentage use of biofuels decreasing as a proportion of overall fuel use due to increased LPG uptake (Pandey et al., 2014). Cow dung cakes remain prevalent as a fuel because they are cheap, readily available, sustainable and ease pressure on local fuel wood resources. Few studies have reported emissions data from cow dung cake

(Venkataraman et al., 2010; Stockwell et al., 2016; Koss et al., 2018; Fleming et al., 2018), leaving considerable uncertainty over the impact that cow dung cake combustion has on air quality. LPG usage has increased from around 100 to 500 million users over the same period, but only reflects around 10% of current rural fuel consumption (Gould and Urpelainen, 2018).

~~Inventories~~India-specific inventories which include residential burning indicate a considerable emission source of total NMVOCs of around 6000-7000 kt yr^{-1} (Pandey et al., 2014; Sharma et al., 2015). Burning is likely to have a large impact on air quality in India, but considerable uncertainties exist over the total amount of NMVOCs released owing to a lack of India specific emission factors and information related to the spatial distribution of emissions.

Few studies exist measuring highly speciated NMVOC emission factors from fuels specific to India. Recent studies using PTR-ToF-MS to develop emission factors, which are more reflective of the range of species emitted from burning, have focussed largely on grasses, crop residues and peat (Stockwell et al., 2015) as well as fuels characteristic of the western U.S. (Koss et al., 2018). A previous study measured emission factors of NMVOCs from cow dung cake using gas chromatography with flame ionisation detection (GC-FID) of 8-32 g kg^{-1} (EPA, 2000). Fleming et al. (2018) quantified 76 NMVOCs from fuel wood and cow dung cake combustion using *chulha* and *angithi* stoves by collecting samples into Kynar bags, transferring their contents into canisters and off-line analysis using GC-FID, GC-ECD (electron capture detector) and GC-MS. The emission factors measured from these 76 NMVOCs were 14 g kg^{-1} for cow dung cake burnt in *chulha* stoves, 27 g kg^{-1} for cow dung cake burnt in *angithi* stoves and 6 g kg^{-1} for fuel wood burnt in *angithi* stoves. An emission factor from one single dung burn measured using PTR-ToF-MS was considerably larger at around 66 g kg^{-1} (Koss et al., 2018). Emissions from dung in Nepal have also been measured (Stockwell et al., 2016) by sampling into whole air sample canisters followed by off-line analysis with GC-FID/ECD/MS and Fourier-transform infrared spectroscopy (FTIR). However, very few speciated NMVOC measurements were made and the emission factors were similar to those measured using just GC (Fleming et al., 2018). Studies have also focussed on making detailed measurements, using a range of techniques, from the burning of municipal solid waste (Christian et al., 2010; Yokelson et al., 2011; Yokelson et al., 2013; Stockwell et al., 2015; Stockwell et al., 2016; Sharma et al., 2019) and crop residues (Stockwell et al., 2015; Koss et al., 2018; Kumar et al., 2018).

Detailed chemical characterisation of NMVOC emissions from fuel types widely used in the developing world is much needed to resolve uncertainties in emission inventories used in regional policy models and global chemical transport models. A greater understanding of the key sources is required to characterise and hence understand air quality issues to allow the development of effective mitigation strategies. In the present study we measure comprehensive emission factors of NMVOCs from a range solid fuels characteristic to northern India.

2. Methods

2.1 Fuel collection and burning facility

A total of 76 fuels, reflecting the range of fuel types used in northern India, were collected from across ~~New~~ Delhi (see Figure 1 and Table 1). Cow dung cake usage was prominent in the north and west regions, whereas fuel wood use was more evenly spread across the state. Municipal solid waste was collected from Bhalaswa, Ghazipur and Okhla landfill sites. Collection also included less used local fuel types which were found being burnt~~,~~ including crop residues, sawdust and charcoal. A low-cost LPG stove, widely promoted across India as a cleaner fuel ~~(Singh et al., 2017), was also purchased to allow~~through government initiatives such as the Pradhan Mantri Ujjwala Yojana and Pratyaksh Hanstantrit Labh schemes, was used for direct emission comparison with other local fuel types.

Fuels were burnt at the CSIR-National Physical Laboratory (NPL), New Delhi, under controlled conditions utilizing a combustion chamber ~~that has been well described previously~~ based on the design of Venkataraman and Rao, (2001). Several previous studies have been based on this chamber design (Venkataraman and Rao, 2001; Venkataraman et al., 2002; Saud et al., 2011; Saud et al., 2012; Singh et al., 2013)~~, using expert local judgement to ensure conditions replicated real world burning conditions. Fuel (200 g) was~~, which was designed to simulate the convection-driven conditions of real-world combustion and is displayed in the Supplementary Information S1. The burn-cycle used in this study was adapted from the VITA water-boiling test, which was designed to simulate emissions from cooking and included emissions from both low- and high-temperature burning conditions. Fuels were collected and stored in the same manner as local customs using expert local judgement. This was designed to ensure that the moisture content of fuel wood samples was like those being burnt locally and that the combustion replicated real-world burning conditions encountered in local cooking practices, which should consequently give a more reflective NMVOC emission factor.

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Fuel (200 g) was placed 45 cm from the top of the hood and, rapidly heated to spontaneous ignition, with emissions convectively driven into a hood and up a flue to allow enough dilution, cooling, and residence time to achieve the quenching typically observed in indoor environments. These conditions have been previously optimised to ensure that emissions entrainment into the hood did not exert a draft which altered combustion conditions. The mid-point velocity of gases driven up the flue by convection was measured by a platinum hot-wire sensor, calibrated for total flow rate using a standard orifice calibrator. Samples were drawn down a sample line at 4.4 L min^{-1} (Swagelok, ¼” PFA, < 2.2 s residence time) from the top of the flue, passed through a pre-conditioned quartz filter (ø = 47 mm, conditioned at 550 °C for 6 hours and changed between samples) held in a filter holder (Cole-Parmer, PFA) which was subsampled for analysis by PTR-ToF-MS, GC×GC-FID and DC-GC-FID instruments at a distance no greater than 5 m from the top of the flue.

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Measurements of *n*-alkanes from *n*-tridecane (C$_{13}$) to eicosane (C$_{20}$) were also made from a subset of ~~30~~29 burns using solid phase extraction disks (SPE, Resprep, C$_{18}$). Samples were passed through a cooling and dilution chamber designed to replicate the immediate condensational processes that occur in smoke particles approximately 5-20 mins after emission, yet prior to photochemistry which may change composition (Akagi et al., 2011). ~~Further details of SPE sample collection are given in~~ (Stewart et al., 2020a)~~.~~Further details of SPE sample collection are given in Stewart et al. (2020a).

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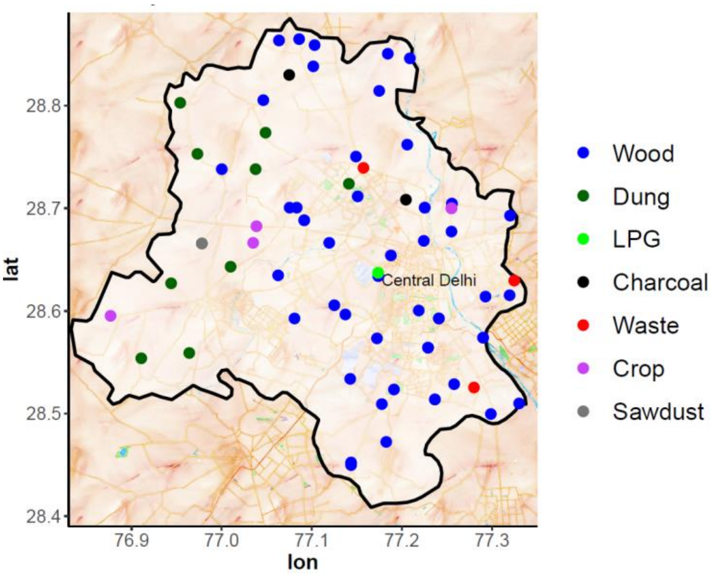

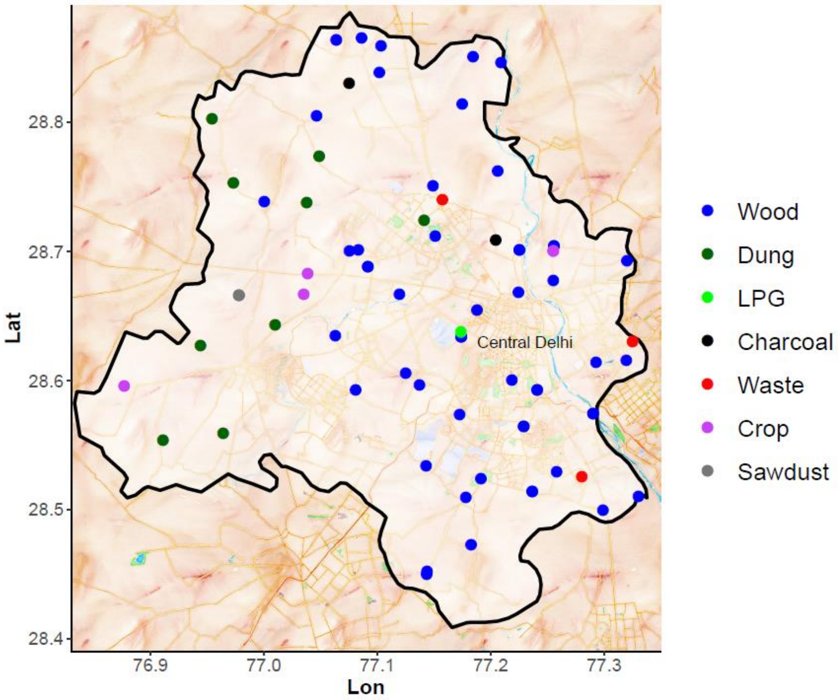

Figure 1. Locations across ~~New~~ Delhi used for the local surveys into fuel use and collection of representative biomass fuels. Map tiles by Stamen Design~~, under CC BY 3.0.~~. Data by © OpenStreetMap~~,~~ contributors 2020. Distributed under ~~CC~~a Creative Commons BY~~–~~SA~~.~~ License.

Table 1. Types and numbers of fuels burnt, the mean emission factor of total measured NMVOCs (TVOC) in g kg^{-1} ~~measured~~ and standard deviation (SD) from all available burns. ~~Discussion of TVOC calculation given is given in the text.~~

Fuel woods	*n*	TVOC	SD	Other	*n*	TVOC	SD
Azadirachta indica	3	18.6	7.9	Cow dung cake	8	61.9	18.4
Morus spp	4	27.4	21.1	*Cocos nucifera*	2	57.4	23.3
Melia azedarach	2	23.7	13.1	Charcoal	2	5.1	3.9
Shorea spp	2	9.8	2.2	Sawdust	2	71.3	60.8
Ficus religiosa	2	51.9	63.4	Waste	3	87.3	31.4
Syzygium spp	2	8.9	4.9	LPG	3	5.8	5.6
Ficus spp	2	7.1	1.2	Cow dung cake mix	1	34.7	-
Vachellia spp	2	13.5	9.7	*Solanum melongena*	2	13.6	6.5
Dalbergia sissoo	2	17.9	8.8	*Brassica spp*	2	41.0	45.5
Ricinus spp	2	8.5	2.5				
Holoptelea spp	2	6.0	0.8				
Mixed woods	1	6.1	-				
Saraca indica	2	12.9	5.2				
Populus spp	1	8.5	-				
Pithecellobium spp	2	19.5	5.4				

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Eucalyptus spp	2	6.9	1.9
Prosopis spp	6	14.5	10.4
Mangifera indica	2	12.4	3.4
Plywood	8	26.6	24.3
Processed wood	2	33.7	17.2

2.2 PTR-ToF-MS

The PTR-ToF-MS (PTR 8000; Ionicon Analytik, Innsbruck) instrument from Physical Research Laboratory (PRL), Ahmedabad was used to quantify 107 masses and subsampled the common inlet line using ¼ inch PFA. Additional details of the PTR-ToF-MS system used in this study are given in previous papers (Sahu and Saxena, 2015; Sahu et al., 2016). The sample air was diluted into zero air, generated by passing ambient air (1 L min^{-1}) through a heated platinum filament at 550 ^{o}C, before entering the instrument with an inlet flow of 250 ml min^{-1}. Samples were diluted by either 5 or 6.25 times (50 ml min^{-1} in 200 ml min^{-1} zero air or 40 ml min^{-1} in 210 ml min^{-1} zero air). The instrument was operated with an electric field strength (E/N, where N is the buffer gas density and E is the electric field strength) of 120 Td. The drift tube temperature was 60 °C with a pressure of 2.3 mbar and 560 V applied across it.

Calibrations were performed twice a week using a gas calibration unit (Ionicon Analytik, Innsbruck). The calibration gas (Apel-Riemer ~~Enironmental~~Environmental Inc., Miami) contained 18 compounds: methanol, acetonitrile, acetaldehyde, acetone, dimethyl sulphide, isoprene, methacrolein, methyl vinyl ketone, 2-butanol, benzene, toluene, 2-hexanone, *m*-xylene, heptanal, α-pinene, 3-octanone and 3-octanol at 1000 ppbv (± 5%) and β-caryophyllene at 500 ppbv (± 5%). This standard was dynamically diluted into zero air to provide a 6-point calibration. The normalised sensitivity (ncps/ppbv) was then determined for each mass using a transmission curve (Taipale et al., 2008). ~~The maximum error in this calibration approach has been shown to be 21%. Peak assignment was assisted with results reported by previous burning studies and references therein (Brilli et al., 2014; Stockwell et al., 2015; Koss et al., 2018), but the~~The maximum error in this calibration approach has been shown to be 21% (Taipale et al., 2008). Peak assignment was assisted with results reported by previous burning studies and references therein (Brilli et al., 2014; Stockwell et al., 2015; Koss et al., 2018). The results may also contain other indistinguishable structural isomers not mentioned here.

Mass calibration and peak fitting of PTR-ToF-MS data were performed using PTRwid software (Holzinger, 2015). Count rates (cps) of each mass spectral peak were normalised to the primary ion (H_3O^+) and water cluster ($H_3O.H_2O$)$^+$ peaks, and mixing ratios were then determined for each mass using the normalised sensitivity. Where compounds known to fragment in the PTR-

ToF-MS were identified, the mixing ratio of these species was calculated by summing parent ion and fragment ion mixing ratios. Before each burn, ambient air was sampled to provide a background for the measurement.

Petrol and diesel fuel samples were collected from an Indian Oil fuel station in Pusa, New Delhi, and the headspace analysed to allow comparison ~~with~~of benzene~~/~~ to toluene ratios. This was designed to analyse the ratios in evaporative emissions, as these have been shown to be an important source of atmospheric NMVOCs (Srivastava et al., 2005; Rubin et al., 2006; Yamada et al., 2015), which for example represented ~ 15% of anthropogenic UK NMVOC emissions in 2018 (Lewis et al., 2020). Fuel samples were placed in a small metal container (¼" Swagelok cap) which was connected to a two-way tap (¼" Swagelok~~) which could be opened and closed.~~). The tap was connected to a t-piece (¼" Swagelok) which had a flow of zero air (250 ml min^{-1}) passed through it and could be sampled by the PTR-ToF-MS. The tap was then opened ~~and closed which allowed~~to analyse the headspace of fuels~~ to be analysed~~.

2.3 DC-GC-FID

Gas chromatography was used to analyse entire burns to provide an integrated picture of emissions from fuel types. The DC-GC-FID sampled 51 burns to measure 19 C_2-C_7 non-methane hydrocarbons (NMHCs) and C_2-C_5 oxygenated ~~VOCs~~NMVOCs (OVOCs) (Hopkins et al., 2003). A 500 ml sample (1.5 L pre-purge of 100 ml min^{-1} for 15 minutes, sample at 17 mL min^{-1} for 30 minutes) was collected (Markes International CIA Advantage), passed through a glass finger at -30 ^{o}C to remove water and adsorbed onto a dual-bed sorbent trap (Markes International ozone precursors trap) at -20 ^{o}C (Markes International Unity 2). The sample was thermally desorbed (250 ^{o}C for 3 minutes) then split 50:50 and injected into two separate columns for analysis of NMHCs (50 m × 0.53 mm Al_2O_3 PLOT) and OVOCs (10 m × 0.53 mm LOWOX with 50 µm restrictor to balance flow). The oven was held at 40 ^{o}C for 5 minutes, then heated at 13 ^{o}C min^{-1} to 110 ^{o}C, then finally at 8 ^{o}C min^{-1} to 200 ^{o}C with a 30-minute hold.

2.4 GC×GC-FID

The GC×GC-FID was used to measure 58 C_7-C_{12} hydrocarbons (C_7-C_{12} alkanes, monoterpenes and monoaromatics) and collected 3 L samples (100 ml min^{-1} for 30 minutes) using an adsorption-thermal desorption system (Markes International Unity 2). NMVOCs were trapped onto a sorbent (Markes International U-T15ATA-2S) at -20 ^{o}C with water removed in a glass cold finger at -30 ^{o}C, removed and heated to ~ 100 ^{o}C after each sample to prevent carryover of unanalysed, polar interfering compounds. The sample was thermally desorbed (250 ^{o}C for 5 minutes) and injected splitless down a transfer line. Analytes were refocussed for 60 s using liquid CO_2 at the head of a non-polar BPX5 held at 50 psi (SGE Analytical 15m × 0.15 µm × 0.25 mm~~)~~), which was connected to a polar BPX50 at 30 psi (SGE Analytical 2 m × 0.25 µm × 0.25 mm) *via*. a modulator held at 180 °C (5 s modulation, Analytical Flow Products ELDV2-MT). The oven was held for 2 minutes at 35 °C, then ramped at 2.5 °C min^{-1} to 130 °C and held for 1 minute with a final ramp of 10 °C min^{-1} to 180 °C and hold of 8 minutes. The GC systems were tested for breakthrough to ensure trapping of the most volatile components (see the Supplementary Information ~~S1 for an example from the GC×GC FID).~~S2). Calibration was carried out using ~~a~~ 4 ppbv gas ~~standard~~standards containing alkanes, alkenes and aromatics ~~(NPL UK)~~purchased from the British National Physical Laboratory and through the relative response of liquid standard injections to toluene for components not in this gas standard, as detailed elsewhere (Dunmore et al., 2015; Stewart et al., 2020b). Integration of peak areas was performed in Zoex GC image software (Zoex, USA). Peaks were individually checked and where peaks were split in the software, they were manually joined. The areas corresponding to alkane isomers were manually joined within the GC image software and calibration performed by comparing the areas to the corresponding *n*-alkane. For both GC instruments, blanks of ambient air were made at the beginning, middle and end of the day and the mean subtracted from samples.

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2.5 ~~GCxGC~~GC×GC-ToF-MS

Measurements were made of a subset of 29 burns of C_{13}-C_{20} alkanes, as well as other gas-phase species to assist with qualification of mases measured by PTR-ToF-MS, by adsorbing samples to the surface of SPE disks with analysis by GC×GC-ToF-MS, as detailed in Stewart et al. (2020a). Samples of 180 L were adsorbed to the surface of C_{18} coated SPE disks (Resprep, C_{18}, 47 mm) prewashed with 2 × 5 mL acetone washes and 1 × 5 mL methanol wash. These samples were collected at 6 L min^{-1} over 30 minutes using a low volume sampler (Vayubodhan Pvt.Ltd) which passed samples through a cooling and dilution chamber at 46.7 L min^{-1}. Samples were then wrapped in foil, placed in an airtight bag, and kept frozen until analysis.

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SPE extracts were spiked with an internal standard~~,~~ (EPA 8270 Semivolatile Internal Standard Mix, 2000 µg mL^{-1} in DCM) and extracted using accelerated solvent extraction into ~~EtOAc.~~ethyl acetate. Extracts were analysed using GC×GC-ToF-MS (Leco Pegasus BT 4D) using a 10:1 split injection (1 µL injection, 4 mm taper focus liner, SHG 560302). The primary dimension column was a RXI-5SilMS (Restek, 30 m × 0.25 µm × 0.25 mm) connected to a second column of RXI-17SilMS (Restek, 0.25 µm × 0.25 mm, 0.17m primary GC oven, 0.1 m

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Peaks assignment was conducted through comparison of retention times to known standards and comparison to the National Institute of Standards and Technology (NIST) mass spectral library. ~~Peaks with no standard available were tentatively identified if the NIST library hit exceeded 700 (Stein, 2011).~~

Integration was carried out within the ChromaTOF 5.0 software package (Leko, 2019). Eight blank measurements were made at the beginning and end of the day by passing air from the chamber (6 L min^{-1} for 30 mins) through the filter holder containing a PTFE filter and an SPE disk. Blank corrections have been applied by subtracting the mean of blank values closest to measurement of the sample. An 8-point calibration was performed for *n*-alkanes using a commercial standard (C_7-C_{40} saturated alkane standard, certified 1000 µg mL^{-1} in hexane, Sigma Aldrich 49452-U) diluted in the range 0.25 – 10 µg ml^{-1}.

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3. Results

~~3.1 Chromatography~~

3.1 Comparison of chromatograms from combustion of different fuel types

Figure 2 shows GC×GC-FID chromatograms obtained from collecting the emissions during the combustion of LPG (Figure 2A), *Saraca indica* fuel wood (Figure 2B), cow dung cake (Figure 2C) and municipal solid waste (Figure 2D). Figure 2D is labelled to show the position of NMVOCs measured and displays a homologous series of *n*-alkanes from *n*-heptane (C_7) to *n*-tetradecane (C_{14}) along the bottom, with the 1-alkenes positioned to the left. Above are more polar species such as monoterpenes, aromatics from benzene to substituted monoaromatics with up to 5 carbon substituents, and at a higher second dimension retention time even more polar species, such as styrene.

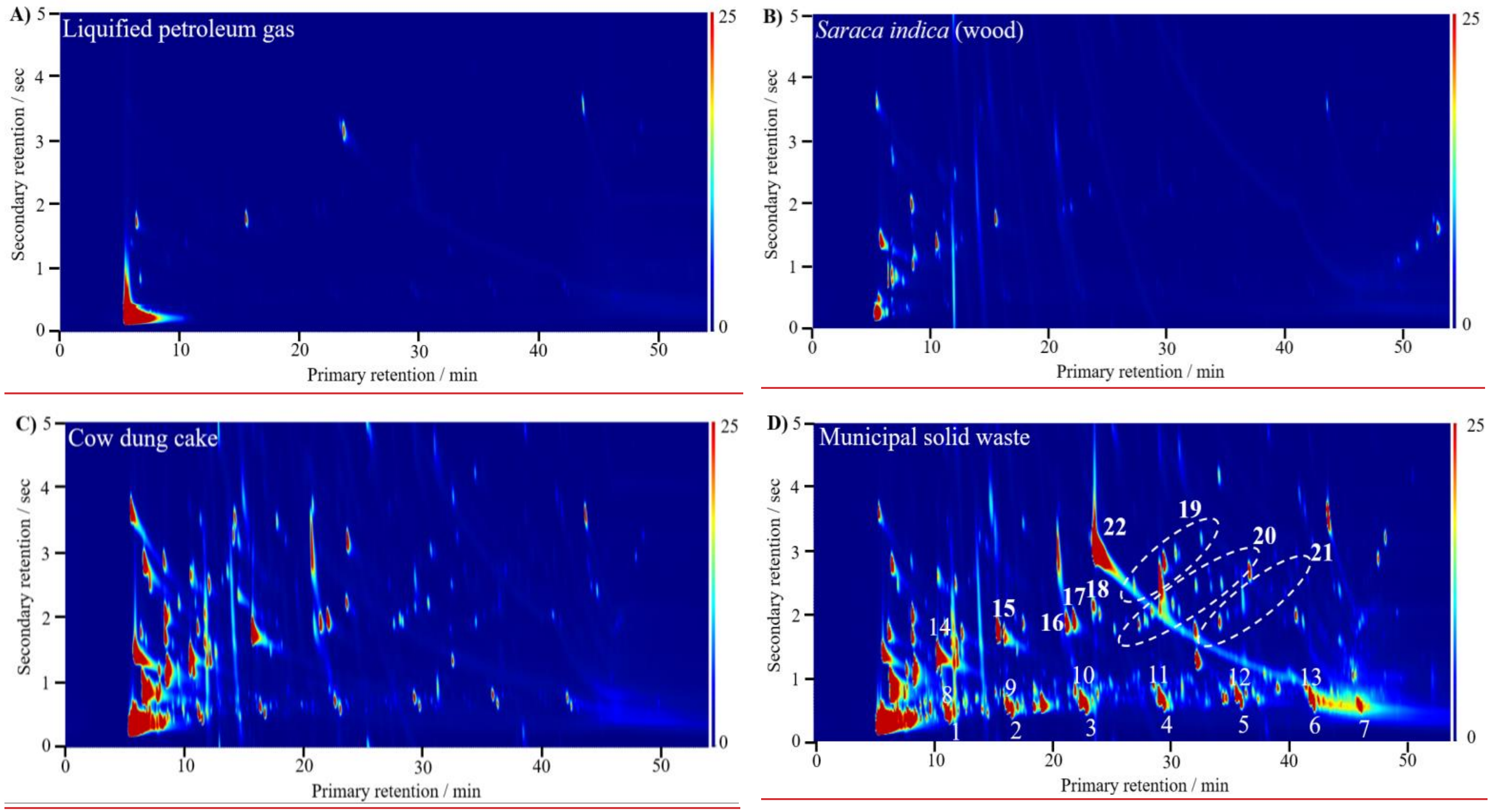

Figure 2. GC×GC-FID chromatograms from burning (A) = LPG, (B) = *Saraca indica* (fuel wood), (C) = cow dung cake and (D) = municipal solid waste samples where 1-7 = *n*-octane – *n*-tetradecane, 8-13 1-octadecene – 1-tridecene, 14 = benzene, 15 = toluene, 16 = ethylbenzene, 17 = *m*/*p*-xylene, 18 = *o*-xylene, 19 = C_3 substituted monoaromatics, 20 = C_4 substituted monoaromatics, 21 = C_5 substituted monoaromatics and 22 = styrene. Samples A-D were collected with the same sample collection parameters and the chromatograms are set at the same contrast level to allow direct comparison between different fuel types.

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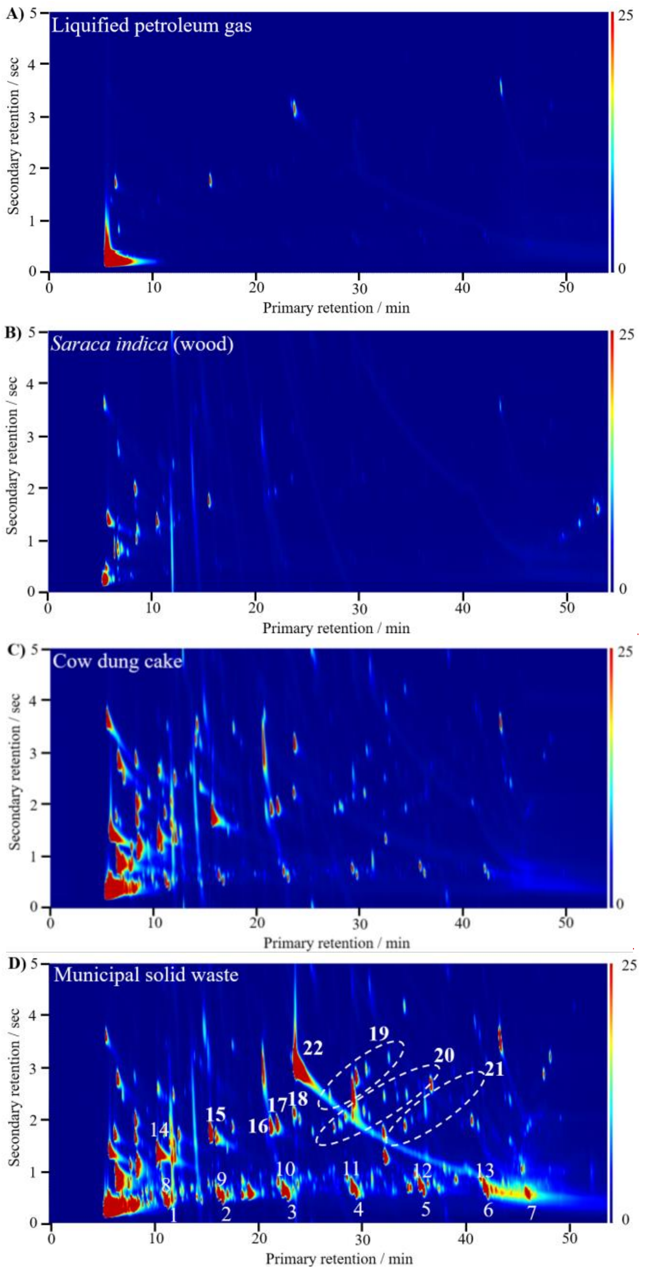
Many peaks were present in the chromatograms for cow dung cake and municipal solid waste, and these fuels released significantly more NMVOCs per unit mass than fuel wood and LPG (see Table 1). Cow dung cake and municipal solid waste released a range of NMVOCs including *n*-alkanes, alkenes, and aromatics. The municipal solid waste (Figure 2D) showed a particularly large and tailing peak 22 owing to large emissions of styrene. Several unidentified peaks were observed in these complex samples which were broad in the second dimension. These were assumed to be from polar, oxygenated species formed during burning such as phenol. These species could not be identified and were not analysed using the GC×GC-FID~~,~~ ~~and peaks~~. Peaks have been omitted if ~~they~~these species were found to interfere ~~with these~~ significantly. Analysis has only been carried out using the DC-GC-FID from ethane (C_2) to *n*-hexane (C_6) owing to the significant presence of coeluting peaks. The large peak in the LPG chromatogram (Figure 2, 1^o ~6 min, 2^o ~ 0.5 s) was from unresolved propane and butane because of the high concentrations from this fuel source.



~~Figure 2. GC×GC-FID chromatograms from burning (A) = LPG, (B) = *Saraca indica* (fuel wood), (C) = cow dung cake and (D) = municipal solid waste samples where 1-7 = *n*-octane – *n*-tetradecane, 8-13 1-octadecene – 1-tridecene, 14 = benzene, 15 = toluene, 16 = ethylbenzene, 17 = *m*/*p*-xylene, 18 = *o*-xylene, 19 = C_3-substituted monoaromatics, 20 = C_4-substituted monoaromatics, 21 = C_5-substituted monoaromatics and 22 = styrene.~~

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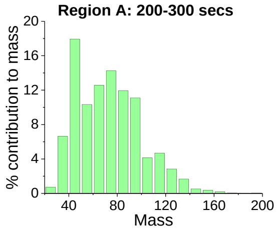
3.2 PTR-ToF-MS concentration time series analysis

Figure 3 shows an example concentration-time series measured by the PTR-ToF-MS for a cow dung cake burn. A sharp rise in NMVOC emissions was seen from the start of the burn which decreased as the fuel was combusted. Emissions of small oxygenated species as well as phenolics and furanics were dominant throughout most of the burn. At the beginning, a greater proportion of lower mass species were released, as shown in the binned mass spectrum of ~~regions~~region A~~/B~~ in Figure 3. At the end in the smouldering phase, emissions were dominated by heavier and lower volatility species (Figure 3, Region ~~C~~B). A previous study indicated larger molecular weight phenolics were from low temperature pyrolysis (Sekimoto et al., 2018).

Figure 4 shows the cumulative mass of species measured from burns of fuel wood, cow dung cake, municipal solid waste, and charcoal as a proportion of the total mass of NMVOCs quantified using PTR-ToF-MS. The results were ~~similar to~~like those reported by Brilli et al. (2014) and Koss et al. (2018): 65-90% of the mass of NMVOCs at emission originated from around 40 NMVOCs, with around 70-90% identification by mass when quantifying around 100 NMVOCs. The largest contributors to the NMVOC mass from burning of fuel wood and cow dung cake were methanol (*m/z* 33.034), acetic acid (*m/z* 61.028) and a peak that reflected the sum of hydroxyacetone, methyl acetate and ethyl formate (*m/z* 75.043). For municipal solid waste samples around 28% of total mass was from methyl methacrylate (*m/z* 101.059) and styrene (*m/z* 105.068), and two of the three municipal solid waste samples released significant quantities of styrene, most likely the result of degradation of polystyrene in the samples.



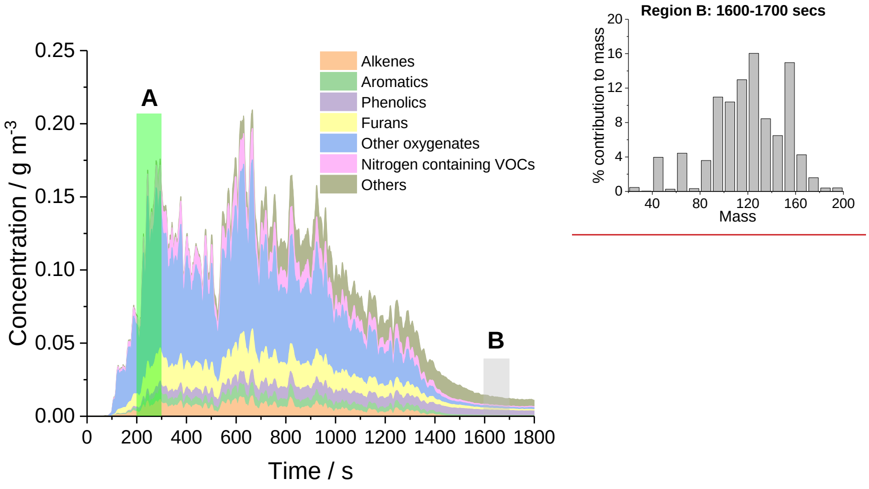


Figure 3. PTR-ToF-MS concentration-time series during the first 30 minutes of a cow dung cake burn coloured by functionality with regions A and B displaying mass spectra placed into m/z bins of 10 Th. Fuel collected from Pitam Pura, Delhi.

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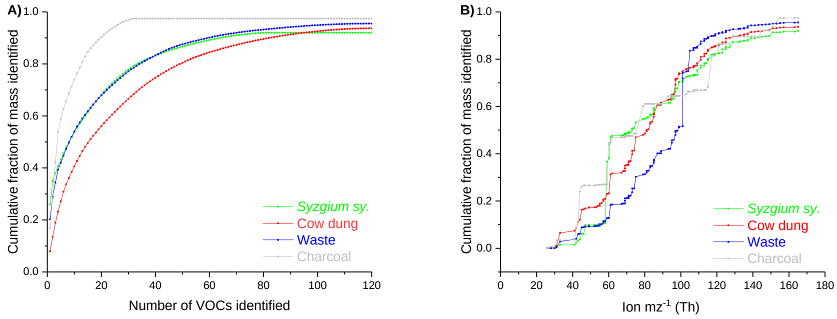

Figure 4. Cumulative NMVOC mass identified from PTR-ToF-MS compared with total NMVOC signal measured by PTR-ToF-MS with (A) ordered by decreasing NMVOC mass contribution and (B) ordered by ion mass. High quantification of emissions from charcoal was due to a low emission factor (2.4 g kg^{-1}).

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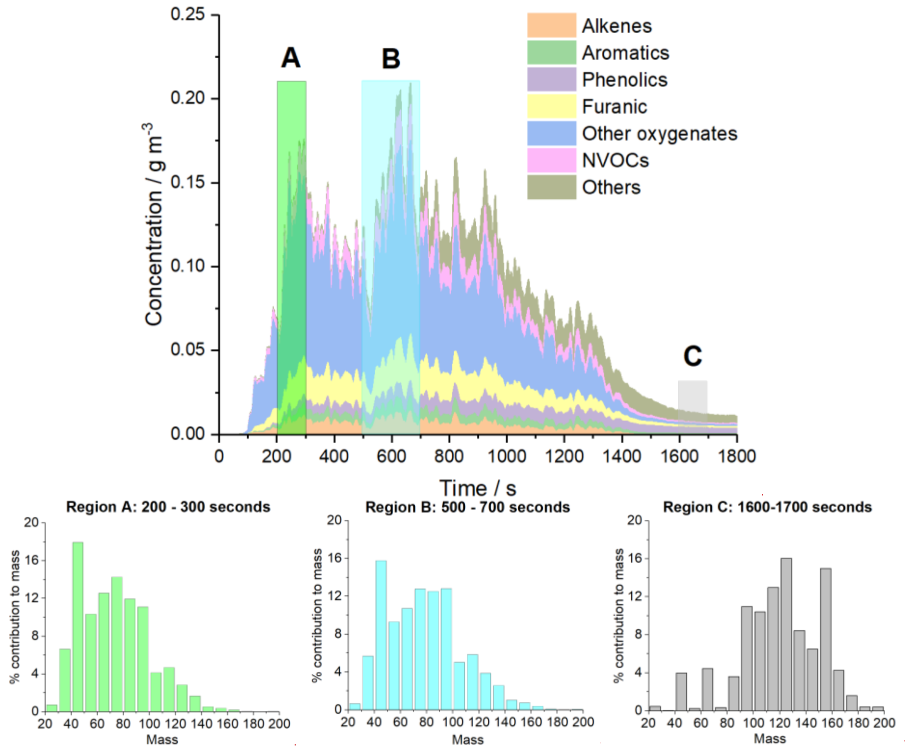
Figure 5 shows a concentration time series for phenolics and furanics from the burning of an example fuel wood. Most species of similar functionality tracked each other. Stockwell et al. (2015) demonstrated that benzene, phenol and furan could act as tracers for aromatic, phenolic and furanic species released from biomass burning. Figure 5A shows that heavier, more substituted phenolics appeared to be released at cooler temperatures. Guaiacol (dark blue) was released at the start of the flaming phase before the temperature increased and more phenol (red) was released at higher burn temperatures. Later in the burn, a larger proportion of vinyl guaiacol (pink) and syringol (yellow) were emitted. This agreed well with previous results which showed that species emitted from lower temperature depolymerisation had a larger proportion of low-volatility compounds compared to higher temperature processes during flaming (Sekimoto et al., 2018; Koss et al., 2018). Figure 5B shows ~~timeseries~~concentration time series of furanic species, with most species showing similar characteristics throughout the burn. The only species to peak later in the burn was 2-hydroxymethyl-2-furan.



~~Figure 3. PTR-ToF-MS concentration-time series during the first 30 minutes of a cow dung cake burn coloured by functionality with regions A, B and C displaying mass spectra placed into m/z bins of 10 Th. Fuel collected from Pitam Pura, New Delhi.~~

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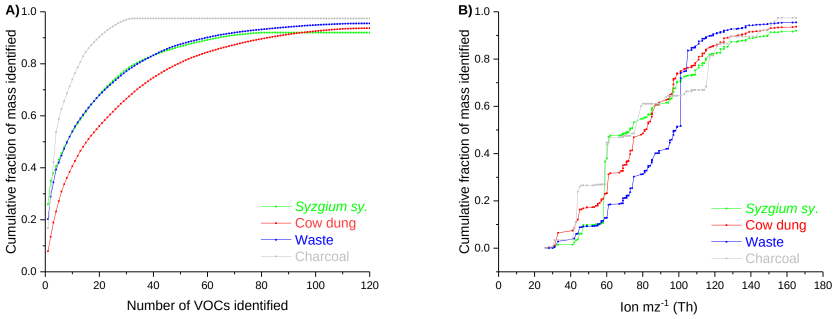

~~Figure 4. Cumulative NMVOC mass identified from PTR-ToF-MS compared with total NMVOC signal from~~

~~PTR-ToF-MS with (A) ordered by decreasing NMVOC mass contribution and (B) ordered by ion mass. High quantification of emissions from charcoal was due to a low emission factor (2.4 g kg^{-1}).~~

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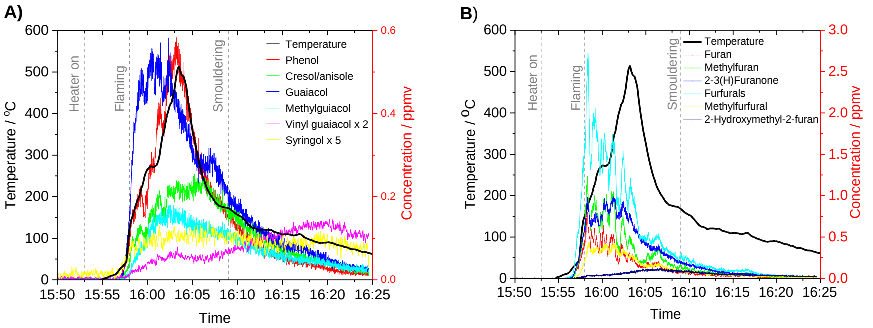

Figure 5. ~~Timeseries~~Concentration time series analysis of phenolic and furanic compounds released from burning of *Azadirachta indica* which released 27.0 g kg^{-1} of NMVOCs. Temperature corresponds to the increase in temperature above ambient measured in the flame directly above the combustion experiment.

3.3 Comparison of emissions data obtained with different instruments

Previous instrument ~~inter-comparisons~~intercomparisons from biomass burning samples were between PTR-MS, GC-MS and open path FTIR (Gilman et al., 2015) and between PTR-ToF-MS, FTIR, ~~broadband~~airborne cavity-enhanced spectroscopy (ACES) and I$^-$-CIMS (Koss et al., 2018). Gilman et al. (2015) showed generally good agreement of slopes of measured emission factors between benzene, ethyne, furan, ethene, propene, methanol, toluene, isoprene and acetonitrile using different instruments/techniques with slopes of ~ 1 ± ~~30~~0.3% and correlation coefficients > 0.9. Koss et al. (2018) showed mean measured values of most NMVOCs from all burns with other instruments compared to the PTR-ToF-MS which agreed within a factor of two and had correlation coefficients > 0.8 for most species except butadienes, furan, hydroxyacetone, furfural, phenol and glyoxal. These previous comparisons ~~underline~~indicate the ~~challenges faced~~level of consistency expected with instrument comparisons of quantitative NMVOC measurements from burning experiments.

Figure 6 shows a comparison of measurements made using the DC-GC-FID, GC×GC-FID and PTR-ToF-MS techniques. Bar plots show that the mean and lower/upper quartiles of all measurements agreed within a factor of two. The correlation coefficient between different instruments is given ~~in~~by blue circles, with all > 0.8. Generally, the mean values measured for the PTR-ToF-MS were slightly larger than using the GC instruments, which was attributed to the presence of other undistinguishable structural isomers measured by the PTR-ToF-MS. Comparison between DC-GC-FID and GC×GC-FID measurements were also complicated by high levels of coelution of additional NMVOC species released from combustion with similar

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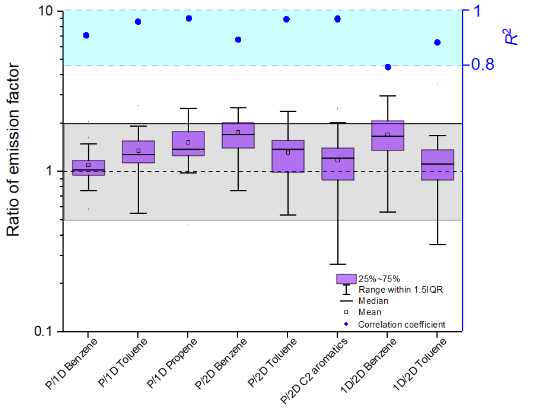
retention times (R_t) to benzene/toluene (R_t = 21/25 mins) on the DC-GC-FID instrument. Generally, the smallest values were measured with the GC×GC-FID instrument, consistent with the greatest ability to speciate isomers and limit the impacts of coelution. Significant efforts were made to synchronise the sample periods for the three instruments as best as possible; however, slight uncertainty existed over the exact time each instrument started measuring when calculating mean sample windows (± 30 s). These factors combined, may help to explain the slight differences observed between different instruments during this study. When multiple instruments have measured the same NMVOC in this study, preference was given to the data from the GC×GC-FID due to the ability of this instrument to resolve coeluting peaks, followed by the DC-GC-FID and then the PTR-ToF-MS.



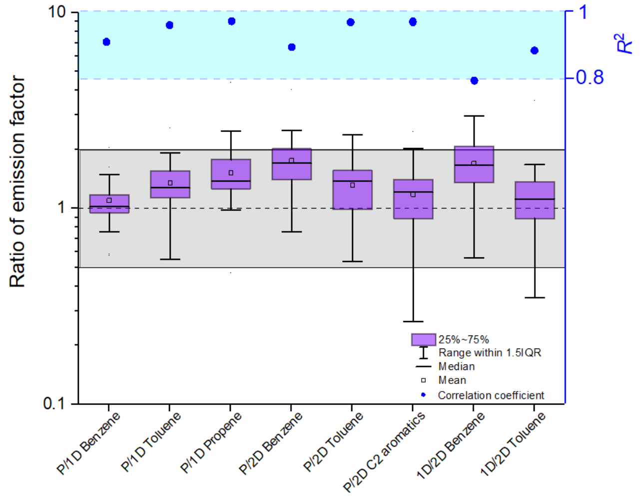


Figure 6. Comparison of PTR-ToF-MS to DC-GC-FID and GC×GC-FID with the black dashed line representing slopes equal to one, grey shaded region = slopes agreeing within a factor of two, shaded blue region indicating correlation coefficients > 0.8 and P = PTR-ToF-MS, 1D = DC-GC-FID and 2D = GC×GC-FID.

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3.4 NMVOC emission factors from biomass fuels

~~Figure 7~~Figure 7 shows a detailed breakdown of the mean NMVOC emission factors by fuel type measured for all 76 burns (see the Supplementary Information ~~S2 for values).~~S3 for values). Emission factors have been determined by calculating the mean NMVOC concentrations up the flue over a 30-minute period, in line with the GC sample time, with any small emissions after this sample window not included. This has been related to the total volume of air convectively drawn up the flue and the mass of fuel burnt (see the Supplementary Information S4 for details). The data is split by functionality to show trends for different chemical types. This shows that burning released a large amount of different NMVOCs, across a wide range of functionalities, molecular weights~~,~~ and volatilities. The large variety of NMVOCs are likely to have different influences on ~~ozone~~O_3 formation, SOA production and the toxicity of emissions.

~~Figure 7~~Figure 7A shows very large emissions of smaller oxygenated species which were driven by methanol, acetic acid and the unresolved combined peak for hydroxy acetone, methyl acetate and ethyl formate. For the fuel wood samples, acetic acid/glycolaldehyde (2.6 g kg^{-1}), methanol (1.8 g kg^{-1}) and acetaldehyde (0.6 g kg^{-1}) compared well with mean values reported by Koss et al. (2018) for pines, firs and spruces (2.7/1.3/1.2 g kg^{-1}) and the mean values measured by Stockwell et al. (2015) mainly from crop residues, grasses and spruces (1.6/1.3/0.~~94~~9 g kg^{-1}). The emission factor from this study for the unresolved peak of hydroxy acetone, methyl acetate and ethyl formate (1.4 g kg^{-1}) was larger than those previously reported by Koss et al. (2018) and Stockwell et al. (2015) of 0.55 and 0.25 g kg^{-1}, respectively.

~~Figure 7~~Figure 7B shows that there were large emissions of furans and furanones from combustion, mainly from methyl furans, furfurals, 2-(3H)-furanone, methyl furfurals and 2-methanol furanone. The World Health Organisation consider furan a carcinogenic species of high-priority ~~(WHO, 2016)~~ with furan and substituted furans~~,~~ suspected to be toxic and mutagenic (Ravindranath et al., 1984; Peterson, 2006; Monien et al., 2011). Furan emissions originate from the low temperature depolymerisation of hemi-cellulose (Sekimoto et al., 2018) and from large alcohols and enols in high-temperature regions of hydrocarbon flames (Johansson et al., 2016). The OH chemistry of furans has been the subject of several studies (Bierbach et al., 1994; Bierbach et al., 1995; Tapia et al., 2011; Liljegren and Stevens, 2013;

Strollo and Ziemann, 2013; Zhao and Wang, 2017; Coggon et al., 2019) and often produces more reactive products such as butenedial, 4-oxo-2-pentenal and 2-methylbutenedial (Bierbach et al., 1994; Gómez Alvarez et al., 2009; Aschmann et al., 2011, 2014). Photo-oxidation of furans may also be a potentially important source of small organic acids such as formic acid (Wang et al., 2020). Oxidation can also occur by nitrate (Berndt et al., 1997; Colmenar et al., 2012) or chlorine radicals (Cabañas et al., 2005; Villanueva et al., 2007). As a result, furans have recently been shown to be some of the species with highest OH reactivity from biomass burning, causing an estimated 10% of the O_3 produced by the combustion emissions in the first 4 hours after emission (Hartikainen et al., 2018; Coggon et al., 2019). Oxidation of furans can lead to SOA production (Gómez Alvarez et al., 2009; Strollo and Ziemann, 2013) with an estimated 8-15% of SOA caused by furans emitted by burning of black spruce, cut grass, Indonesian peat and ponderosa pine and 28-50% of SOA from rice straw and wiregrass (Hatch et al., 2015), although SOA yields are still uncertain for many species (Hatch et al., 2017).

Phenols are formed from the low-temperature depolymerisation of lignin (Simoneit et al., 1993; Sekimoto et al., 2018) which is a polymer of randomly linked, amorphous high-molecular weight phenolic compounds (Shafizadeh, 1982). Owing to their high emission ratios and SOA formation potentials, phenolic compounds contribute significantly to SOA production from biomass-burning emissions (Yee et al., 2013; Lauraguais et al., 2014; Gilman et al., 2015; Finewax et al., 2018).

~~Figure 7~~Figure 7C shows that the largest phenolic emissions from fuel wood in this study were methoxyphenols, with significant contributions from phenol, guaiacol, cresols and anisole. Phenolic emissions from sawdust were dominated by guaiacol and creosol. Phenolic emissions from coconut shell were greatest, most likely as a result of the lignin rich nature of coconut shell (Pandharipande, 2018). The larger mean emission of ~~furans~~furanics (3.2 g kg^{-1}) compared to ~~phenols~~phenolics (1.1 g kg^{-1}) from fuel wood was consistent with wood being composed of around 75% cellulose/hemicellulose and 25% lignin (Sjöström, 1993).

~~Figure 7~~Figure 7D shows that the largest alkene emission was styrene from burning municipal solid waste, likely caused by the presence of polystyrene in the fuel. Emissions of alkenes from fuel woods were dominated by ethene and propene, species with high photochemical ozone creation ~~potential~~potentials (Cheng et al., 2010). Monoterpenes¸ which are extremely reactive with the OH radical (Atkinson and Arey, 2003), were emitted from combustion of sawdust, cow dung cake and municipal solid waste samples.

Ethane and propane dominated the alkane emissions for fuel wood samples (see ~~Figure 7~~Figure 7E). A wider range of alkanes from C_2-C_{20} were observed from combustion of coconut, cow dung cake and municipal solid waste. The largest alkane emission by mass was from LPG due to unburnt propane and butane.

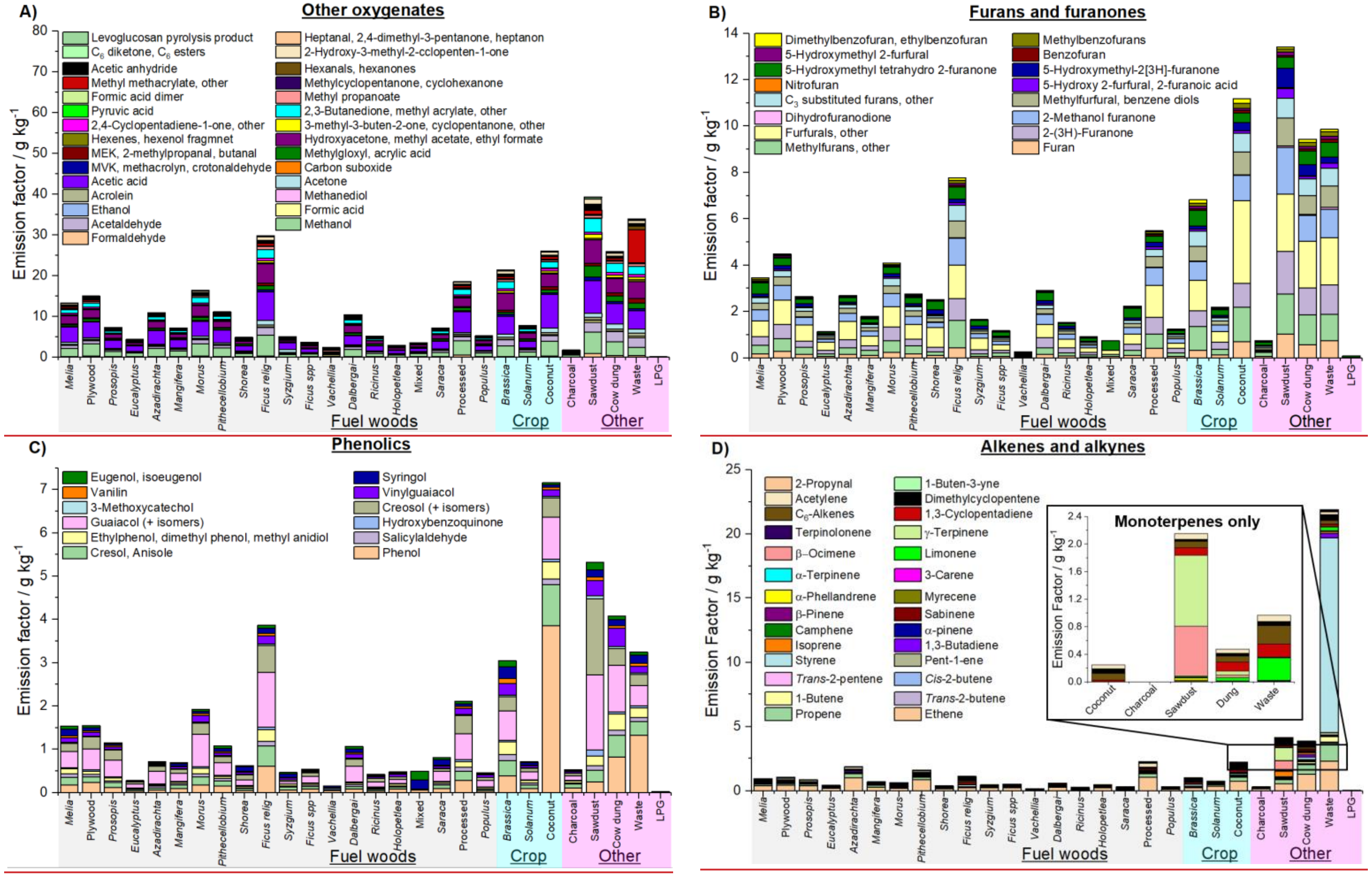

Figure 7. Measured emission factors grouped by functionality.

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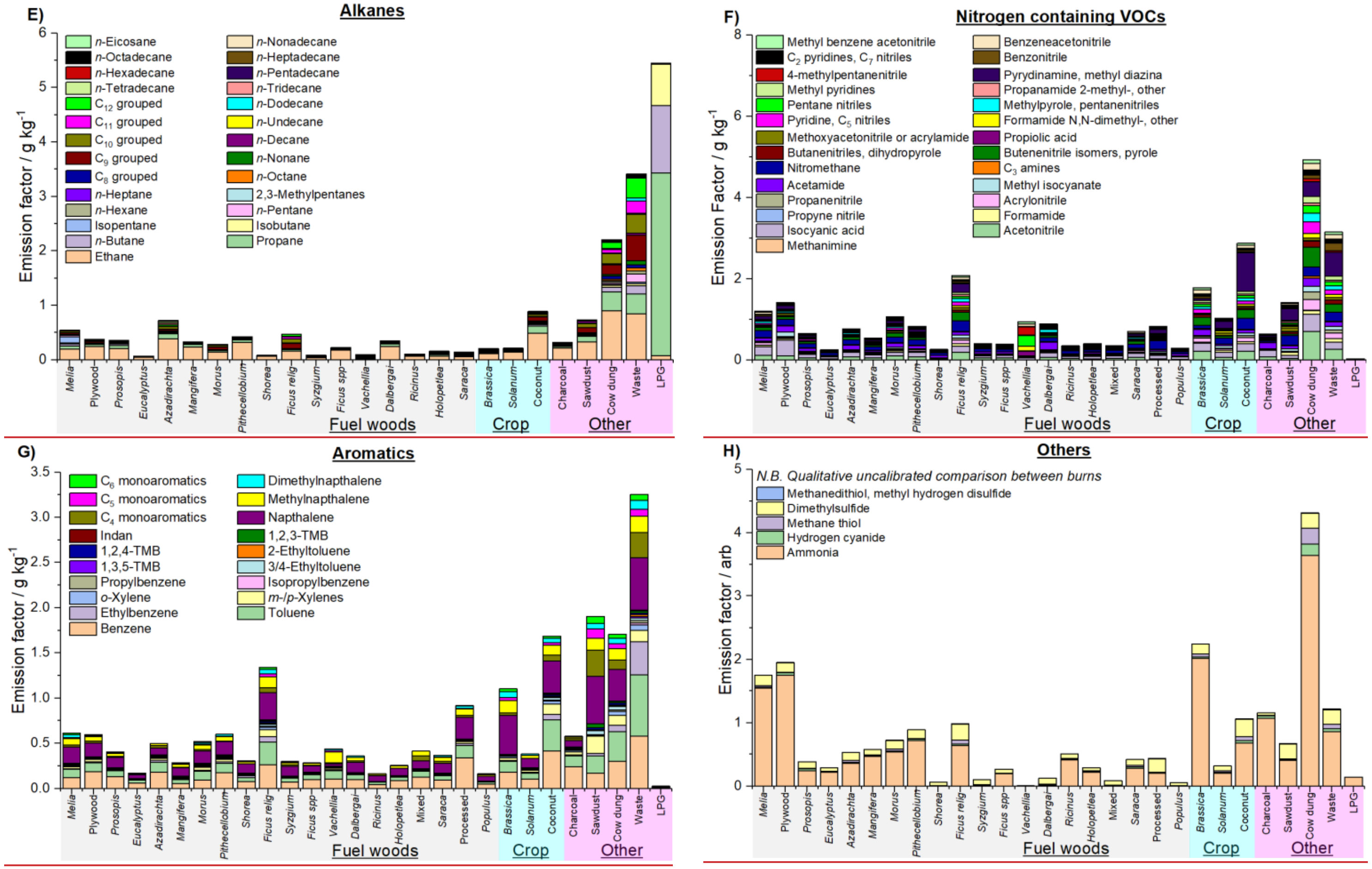

Figure 7 continued.

Nitrogen containing ~~VOCs (NVOCs)~~NMVOCs are formed from the volatilisation and decomposition of nitrogen-containing compounds within the fuel, mainly from free amino acids but can also be from pyrroline, pyridine and chlorophyll (Leppalahti and Koljonen, 1995; Burling et al., 2010; Ren and Zhao, 2015). ~~NVOCs~~Nitrogen containing NMVOCs are of interest because nitrogen may be important in the development of new particles (Smith et al., 2008; Kirkby et al., 2011; Yu and Luo, 2014) which act as cloud condensation nuclei (Kerminen et al., 2005; Laaksonen et al., 2005; Sotiropoulou et al., 2006) and alter the hydrological cycle by forming new clouds and precipitation (Novakov and Penner, 1993). They can also contribute to light-absorbing brown carbon (BrC) aerosol formation, effecting climate (Laskin et al., 2015). Additionally, ~~NVOCs~~nitrogen containing NMVOCs can be extremely toxic (Ramírez et al., 2012, 2014; Farren et al., 2015). Cow dung cake was the largest emitter of ~~NVOCs~~nitrogen containing NMVOCs (4.9 g kg^{-1}), releasing large amounts of acetonitrile and nitriles, likely to have a large impact on the toxicity and chemistry of emissions (see ~~Figure 7F).~~

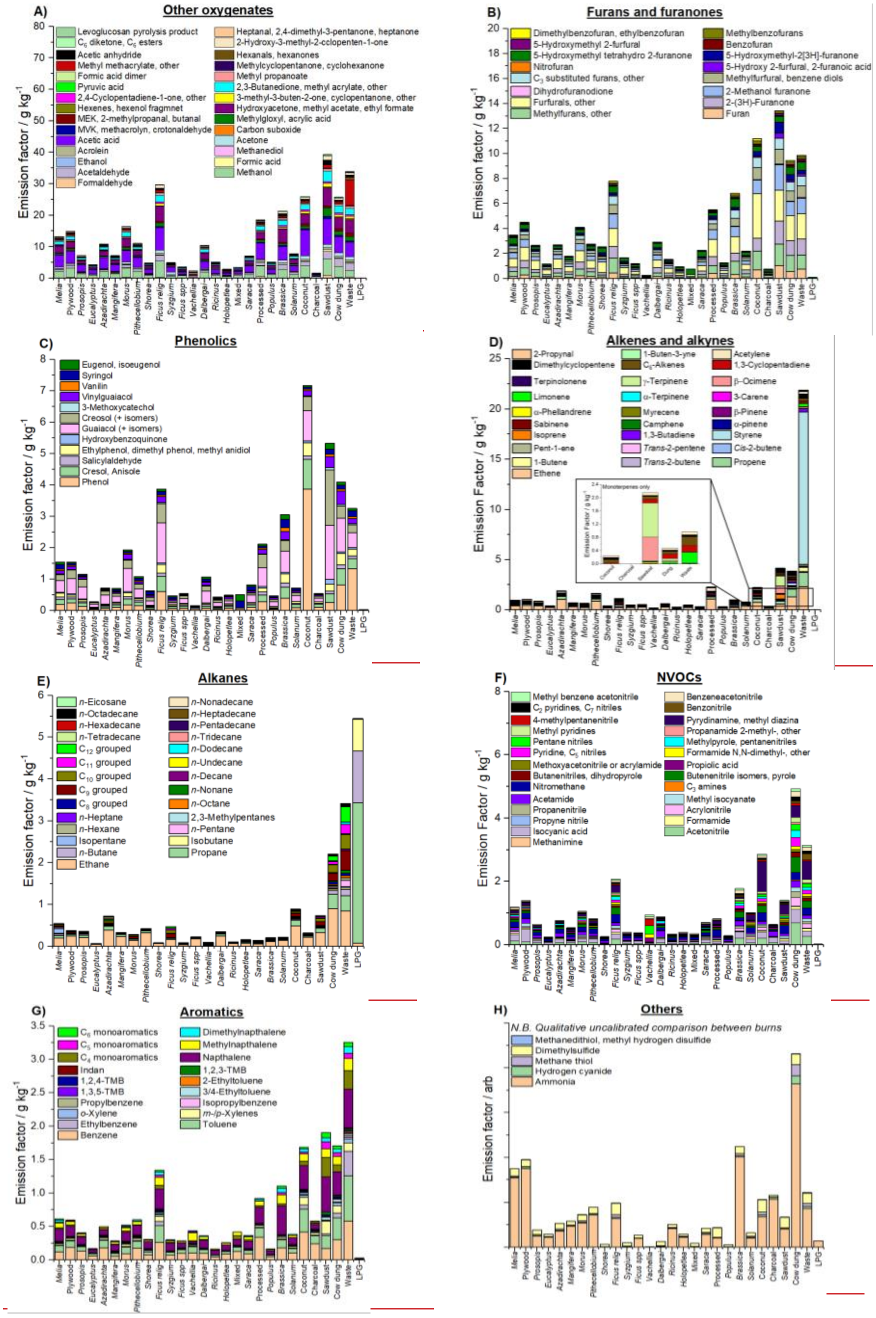

Figure 7F).

Figure 7~~Figure 7. Measured emission factors grouped by functionality.~~

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~~Figure 7~~G shows emissions of aromatics from fuel wood, cow dung cake and municipal solid waste were principally benzene, toluene and naphthalenes. Large emissions of benzene were unsurprising as biomass burning is the largest global benzene source (Andreae and Merlet, 2001). Emissions of benzene, toluene, ethylbenzene and xylenes (BTEX) from cow dung cake (0.5-1.7 g kg^{-1}) were in line with previous measurements of 1.3 g kg^{-1} (Koss et al., 2018) and 1.8 g kg^{-1} (Fleming et al., 2018) but lower than the 4.5 g kg^{-1} reported from cow dung cake combusted from Nepal (Stockwell et al., 2016). Emissions of BTEX from municipal solid waste burning (0.9– 2.6 g kg^{-1}) were comparable to that measured previously (3.5 g kg^{-1}) (Stockwell et al., 2016).

~~Figure 7~~Figure 7H shows a qualitative comparison of species such as ammonia, HCN and dimethyl sulphide which were measured during experiments, but could not be accurately quantified as their sensitivity was too different from the NMVOCs used to build the transmission curve. Cow dung cake emitted significantly more of these species than other fuel types.

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Table 2 shows the total measured emission factors of NMVOCs for different fuel types. ~~These have been determined by calculating the total volume of air convectively drawn up the flue and relating this to the mass of fuel burnt (see the Supplementary Information S3 for details). Emission factors have been calculated over a 30 minute period, in line with the GC sample time, with any small emissions after this sample window not included.~~ The total measured emission factor has been calculated as the sum of the PTR-ToF-MS signal, excluding reagent ion peaks (< *m*/*z* 31 Th~~)~~), water cluster peaks (*m*/*z* 37 Th) and isotope peaks identified for all masses (SIS, 2016). The emission factors for all alkanes and alkenes measured by the GC instruments were also included, as alkanes up to *n*-hexane had proton affinities less than water and larger alkanes had proton affinities similar to water (Ellis and Mayhew, 2014; Wróblewski et al., 2006). This low sensitivity meant that no peaks were present in the PTR-ToF-MS spectra for these larger species. Any alkenes measured by the DC-GC-FID were excluded from the PTR-ToF-MS data. Further information on the species included in the calculation of the total measured emission factor is given in the Supplementary Information ~~S4~~S5.

Table 2. Mean total measured NMVOC emission factors (g kg^{-1}, including IVOC fraction) where high/low EF represent the largest/smallest emission factor measured for a given sample type (g kg^{-1})

and IVOC is the sum of emission factors of species with a mass greater than benzaldehyde (g kg^{-1}) where *n* = number of measurements made.

	Wood	**Dung**	**Waste**	**LPG**	**Charcoal**	**Sawdust**	**Crop**
NMVOC	18.7	62.0	87.3	5.7	5.4	72.4	37.9
High EF	96.7	83.0	119.1	9.8	7.9	114.0	73.8
Low EF	4.3	35.3	56.3	1.9	2.4	28.3	8.9
IVOC	3.~~4~~5	12.6	13.2	0.2	1.4	~~18~~16.9	8.0
n	51	8	3	3	2	2	6

Coconut shell, sawdust, cow dung cake and municipal solid waste released the greatest mass of NMVOC per kg of fuel burnt. The mean emission factor for all fuel woods (18.7 g kg^{-1}) was comparable to that for chaparral (16.6 g kg^{-1}) measured using PTR-ToF-MS by Stockwell et al. (2015). This may be due to similarities between north Indian fuel wood types with chaparral, which is characterised by hot dry summers, and mild wet winters. The mean fuel wood emission factor was smaller than Stockwell et al. (2015) reported for coniferous canopy (31.0 g kg^{-1}). The NMVOC emission measured for cow dung cake (62.0 g kg^{-1}) was comparable to that previously reported (66.3 g kg^{-1}) in literature using PTR-ToF-MS (Koss et al., 2018), but 2-3 times larger than that measured by GC-FID/ECD/MS likely due to those techniques missing significant amounts of emissions (Fleming et al., 2018). Whilst the total measured emissions reported by Fleming et al. (2018) might therefore be an underestimate, it is noteworthy that the emission factors measured by Fleming et al. (2018) in *angithi* stoves for cow dung cake were ~ factor of 4 greater than fuel wood under the same conditions. This result was comparable to this study which showed that cow dung cake emissions were ~ factor of 3 larger than fuel wood, ~~however~~but the techniques used here targeted a greater proportion of total emissions. Moreover, Fleming et al. (2018) reported emission factors from combustion of biomass fuels from a neighbouring state, Haryana, and there may be slight heterogeneity between the different fuels collected in both studies. Venkataraman et al. (2010) and Koss et al. (2018) also showed NMVOC emissions from dung combustion to be greater than from fuel wood. NMVOC emissions from municipal solid waste (87.3 g kg^{-1}) were significantly larger than the ~~7.1~~~ 9 g kg^{-1} (Stockwell et al., 2015) and ~~33.8~~~ 35 g kg^{-1} (Stockwell et al., 2016) previously reported. This was likely due to differences in composition and moisture content of the fuels collected from Indian landfill sites for the present study, compared with the daily mixed waste and plastic bags collected at the US fire services laboratory~~ and plastic bags~~ (Stockwell et al., 2015) and a variety of mixed waste and plastics collected from around Nepal (Stockwell et al., 2016). It seems noteworthy that combustion experiments of fuels collected from developing countries in Stockwell et al. (2016) had larger emission factors than those collected from, and burnt at a laboratory (Stockwell et al., 2015). The mean crop residue combustion emission factor (37.9 g kg^{-1}) was comparable to that reported by Stockwell et al. (2015) (36.8 g kg^{-1}), despite the small number of samples in this study and compositional differences.

Considerable uncertainties exist in consumption estimates for fuels such as cow dung cake and municipal solid waste in India. A previous study estimated that in 1985 in India fuel wood consumption was 220 Tg and cow dung cake consumption 93 Tg (Yevich and Logan, 2003). A different study made an India-wide estimate for 2000 which estimated fuel wood consumption to be 281 (192-409) Tg and cow dung cake consumption to be 62 (35-128) Tg (Habib et al., 2004). A more recent study estimated fuel wood usage at 256 Tg and cow dung cake consumption at 106 Tg for 2007 (Singh et al., 2013). Estimates of the amount of municipal solid waste burnt in India are even fewer than for cow dung cake consumption. Two previous studies have estimated that 81.4 Tg of municipal solid waste was burnt in India in 2010 (Wiedinmyer et al., 2014) and that 68 (45-105) Tg was burnt in 2015 (Sharma et al., 2019). The mean emission factors for cow dung cake and municipal solid waste combustion were considerably larger than for fuel wood and highlight that at an India-wide level these may represent significant NMVOC sources.

Intermediate-volatility organic compounds (IVOCs) are defined as having effective saturation concentration, C^*, $=300\text{-}3\times10^{6}$ μg m^{-3} (Donahue et al., 2012). The C^* of several species was estimated using a previously established approach (Lu et al., 2018), with the IVOC boundary defined in this study at benzaldehyde (m = 106.12) for which C^* was ~ 7×10^{6} μg m^{-3}. Table 2 also shows an approximation for the mean amount of IVOCs released by fuel type. ~~The IVOC fraction has been approximated by considering all NMVOCs with a mass greater than benzaldehyde to be IVOCs.~~ This approach was approximate as vapour pressures depend on both mass and functionality. The fuels tested in this study showed that mean emissions of IVOC species represented approximately 18 – 27% of total measured emissions from all fuel types other than LPG. ~~This agreed well with the IVOC fraction reported by Stockwell et al. (2015) of ~ 14-26%.~~ This demonstrated that ~~biomass burning~~domestic solid fuel combustion is potentially a large global source of IVOCs. In addition, this may represent an underestimate because the quartz filter placed on the sample line may remove IVOC species which have partitioned to the aerosol phase due to the high aerosol concentrations present during source testing. Further studies are required to better understand the contribution of IVOC emissions from biomass burning to SOA formation.

~~Figure 8~~Figure 8A shows the distribution of total measured NMVOC emission factors for fuel wood, cow dung cake, crop residues and MSW. Boxplots show the mean, median, interquartile range and range within 1.5IQR. The solid circles display the spread of measured emission factors by fuel type. The zoomed green region given in Figure 8B specifically focuses on the variability in emission factors of individual species of fuel wood, which has been explored in detail due to the large number of samples. Repeat samples collected from the same location are shaded in grey. For fuel wood, measured NMVOC emission factors varied by over a factor of 20 between 4.3-96.7 g kg^{-1}. The NMVOC emission factors showed a right skewed distribution with a median of 11.7 g kg^{-1}, mean of 18.7 g kg^{-1} and an interquartile range of 15.3 g kg^{-1}. For repeat measurements of identical species of fuel wood collected at the same location, except for *Ficus religiosa*, measured emission factors from repeat experiments varied over a much smaller range, by up to a factor of 2.3. Variation between emissions from these samples were likely due to different moisture contents of actual samples measured and the specific combustion conditions of individual burns. The large variation observed for *Ficus religiosa* was likely due to the samples being significantly different in terms of composition. Despite the samples for *Holopetlea spp* and *Eucalyptus spp* coming from different locations, emission factors for these samples were quite reproducible and only varied by a factor of 1.2-1.5. For remaining identical species of fuel wood collected from different locations, emission factors varied over a much larger range by factors of ~ 2-9.

For the crop residue species studied here, NMVOC emissions were right skewed with a with a median of 29.5 g kg^{-1} which was less than the mean of 37.9 g kg^{-1} and varied from 8.9-73.8 g kg^{-1} with an interquartile range of 53.9 g kg^{-1}. *Cocos nucifera* and *Solanum melongena* were repeat measurements of fuel collected from the same location and varied by factors of 1.8-2. NMVOC emissions from *Brassica spp* fuel, which was collected from different locations, varied by a factor of ~ 8. Cow dung cake and MSW samples were all collected from different locations and varied by up to factors of up to 2.4 and 2.1, respectively.

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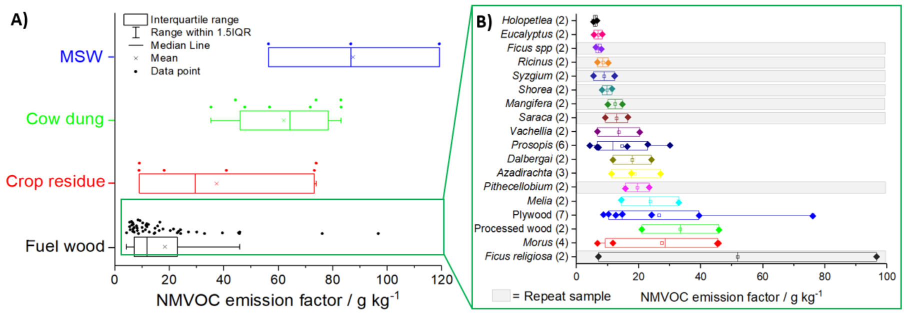

Figure 8. Variability in NMVOC emission factor by fuel type. A) = Range of emission factors measured for fuel wood, cow dung cake, crop residue and municipal solid waste samples with box plots showing the mean, median, interquartile range, range within 1.5IQR and solid circles showing the spread of measured emission factors by fuel type. B) = Zoomed green region displaying range of NMVOC emission factors measured for individual species of fuel wood with grey shaded region indicating repeat samples from the same sample collection location and diamonds indicating the measured NMVOC emission factors.

Figure 9A shows the mean total emissions measured in this study for different fuel types split by functionality. Large variability in total measured emissions were observed for fuel woods, with emission factors from individual burns varying by ~ factor 20. Figure 9B shows the mean emissions by functionality as a proportion of total measured emissions averaged by overall fuel type. Oxygenates were the largest emission (33-55%), followed by furanic compounds (16-21%), phenolics (6-12%) and aromatics (2-9%) for all fuel types except LPG. LPG emissions were mainly alkanes, with a small emission of furanic species. These have previously been reported to be produced in hydrocarbon flames (Johansson et al., 2016).

Figure 9A-B also show the amount of NMVOC which remained unidentified (black). On average 94% of all measured NMVOCs emitted across all burns were ~~quantified. Quantification~~speciated. Speciation was greater than 90% for all sample types, except *Vachellia spp* due to several large unidentified peaks (see the Supplementary Information ~~S5~~S6). Mean ~~quantification~~speciation by fuel type was between 93-96% for all other fuels, except LPG where ~~quantification~~speciation was > 99%.

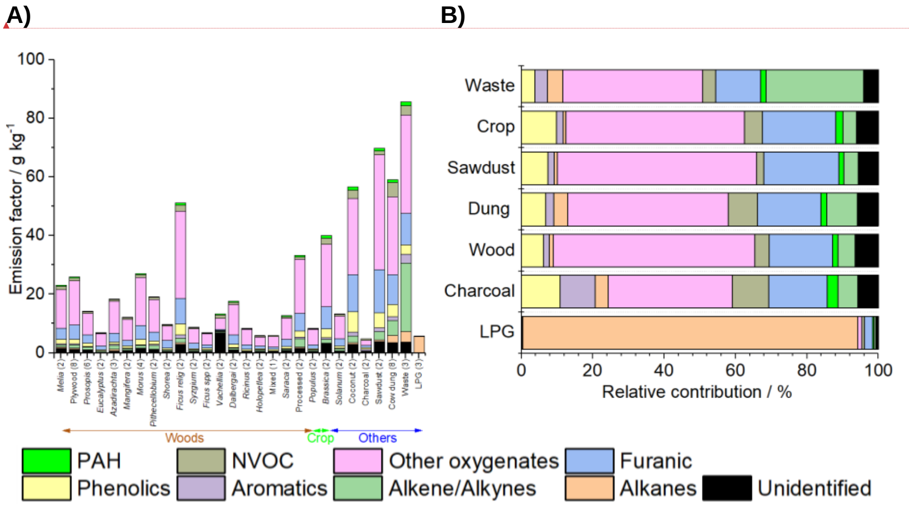

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Figure 9. NMVOC emissions from burning sources in~~ New~~ Delhi, India grouped by functionality with unidentified emissions given by the total NMVOC signal measured by the PTR-ToF-MS minus the fraction quantified using DC-GC-FID, ~~GCxGC~~GC×GC-FID, GC×GC-ToF-MS and PTR-ToF-MS instruments with (A) all fuel types and (B) mean values by type of fuel.

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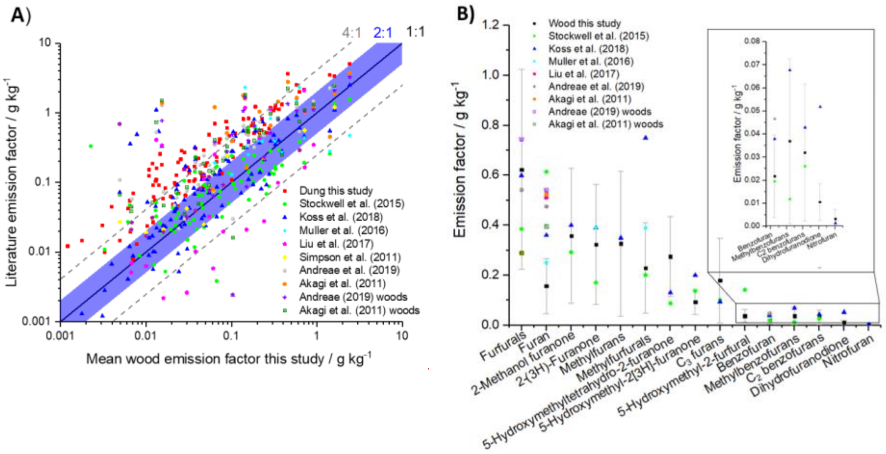
~~The emission factors measured in this study were compared to mean values measured in other studies using PTR-ToF-MS (see Figure 9) for fuel wood, straw, peat and cow dung cake (Koss et al., 2018); grasses, straws and peat (Stockwell et al., 2015) and forest fires (Simpson et al., 2011; Müller et al., 2016; Liu et al., 2017). They were also compared to mean values calculated from reviews for savannah, boreal forest, tropical forest, temperate forest, peatland, chaparral and open cooking (Akagi et al., 2011) and savannah, tropical forest, temperate forest, boreal forests, peat fires and biofuels without fuel wood (Andreae, 2019). Comparison was also made to reviews for mean emission factors from just fuel woods from savannah, boreal forest, tropical forest and temperate forest (Akagi et al., 2011; Andreae, 2019).~~

~~Figure 9A shows that emission factors measured in this study and those measured by Stockwell et al. (2015), Koss et al. (2018), Muller et al. (2016) and Simpson et al. (2011) were generally within a factor of 2-4. The differences in emission factors were likely due to differences in composition between fuels collected from different locations. The emission factors measured in this study were generally smaller than those reported in reviews by Akagi et al. (2011) and Andreae (2019), despite the total NMVOC emission in this study being greater due to measurement of a much wider range of NMVOCs. Emission factors for cow dung cake~~

~~measured in this study were closer to the 4:1 line, which showed that cow dung cake was consistently more polluting per mass burnt than fuel wood.~~



~~Figure 9. Emission factor comparison between this study and literature for (A) all species reported in reviews and comparable studies and (B) furanic species from fuel woods. See text for discussion of fuel types included in each study.~~

~~Figure 9B shows a comparison of emission factors for furanic species from fuel woods compared with those from literature, which showed relatively good agreement within the standard deviation observed from this study. A few notable exceptions were that the emission factor for furfural measured by Muller et al. (2016) was considerably higher (2.3 g kg^{-1}) than this study (0.7 g kg^{-1}), or previous studies, and not included in Figure 9B. The mean emission factor for furan measured in this study was ~ factor 2 lower than other studies. Koss et al. (2018) measured ~ factor 3 higher methyl furfural, ~ 9 higher 5-hydroxymethyl-2-furfural and ~ factor 3 higher dihydrofurandione and Stockwell et al. (2015) measured a higher emission factor than this study of 5-hydroxymethyl-2-furfural by ~ factor of 4.~~

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3.5 Emission ratios

The ratio of the mixing ratios of NMVOCs in the emitted gas can be a useful indicator of their source(s) in ambient air. Ratios can be specific to sources and can allow one source to be distinguished from another. The ratio of *i*-/*n*-pentane can be a useful indicator of whether emissions are anthropogenic or from biomass burning, with a ratio 2.2-3.8 indicative of vehicular emissions, 0.8-0.9 for natural gas drilling, 1.8-4.6 for evaporative fuel emissions and < 1 from burning (Stewart et al., 2020b). Benzene/toluene ratios can also be useful and have been reported from traffic exhaust to be around 0.3 (Hedberg et al., 2002).

i-/*n*-Pentane indicator ratios have been evaluated for fuel wood sources, propane/butane ratios for LPG and benzene/toluene ratios for fuel wood and cow dung cake (see Figure 10). The range of values for multiple different burns have been evaluated rather than just reporting mean and median ratios. The median of *i*-/*n*-pentane ratios from biomass samples measured during this study was ~ 0.7 (see Figure 10). The mean ratio was ~ 1.0, with an interquartile range (IQR) ~ 0.5-1.5, which suggests caution is required when assigning burning sources based on emission ratios due to considerable variability. Despite this, the ratio from solid fuel combustion sources was often less than expected from petrol emissions. The mean ratio of propane/butane from LPG burning was measured to be 3.1. The ratios of benzene/toluene varied considerably between different sources and was measured for fuel wood combustion (2.3), cow dung cake combustion (0.94), petrol liquid fuel (0.40) and diesel liquid fuel (0.20). The range of benzene/toluene ratios for fuel wood was large, with an IQR of ~ 1.5~~-~~- 2.8 and the range within 1.5 IQR shown by the whiskers in Figure 10 from ~ 0.9~~–~~-4.2. Despite the variability of ratios from specific source types, the considerable range of benzene/toluene ratios could potentially be a useful indicator of the origin of unaged (fresh) ambient emissions in ~~New~~ Delhi. However, further study would be required to assess if these ratios were also true in the exhaust of petrol and diesel vehicles in India, or just limited to fugitive emissions. These findings agree well with literature which report mean benzene/toluene ratios of 1.4-5.0 from fuel wood and 0.3 from automotive emissions (Hedberg et al., 2002), indicating that on average biomass burning releases a greater molar ratio of benzene than toluene when compared to automotive emissions.

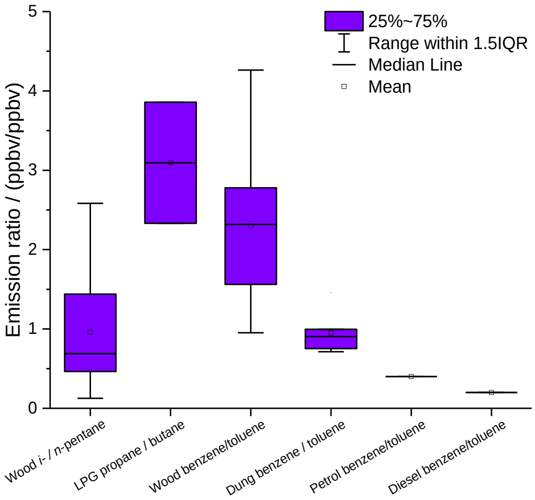

Figure 10. Summary of ratios of NMVOCs measured during this study from the burning of fuel wood, LPG and cow dung cake and from the headspace of liquid petrol and diesel fuels collected in India. The different mean and median values have been considered to evaluate the ratios at emission of specific sources.

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4. ~~Conclusion~~Conclusions

This study was based on comprehensive measurements of NMVOC emissions using a range of detailed and complementary techniques across a large range of functionalities and volatilities. It presented detailed burning emission factors for different NMVOCs from a range of fuels used in ~~New~~the Delhi~~,~~ area of India for residential combustion. This work allowed for a better understanding of the impact of residential combustion on air quality and showed that fuel wood, cow dung cake and municipal solid waste burning sources released significantly more NMVOCs than LPG.

A range of areas where future studies are required to better improve and understand emissions from burning have been highlighted:

1. Better understanding of stove burn conditions on emissions

The impact of stove conditions on NMVOC emissions remains poorly understood. Experiments in this study were carried out using expert local judgment to attempt to ensure that laboratory conditions reflected real-world burning conditions. A range of stoves are used in India for combustion of local fuels, such as *chulha* and *angithi* stoves, and an evaluation of the impact of these on emissions and their relative use and spatial distribution requires further study.

2. Better understanding of the effect of moisture content on modified combustion efficiency

Fuels in this study were collected and stored in a manner designed to be reflective of local practices to ensure that laboratory combustion conditions, and in turn emissions, reflected local burning practices. Future studies should conduct detailed compositional analysis of fuel types and moisture content prior to burning. These studies should also measure CO and CO_2 to allow an evaluation of the impact of modified combustion efficiency on emissions from different fuel types.

3. Limited measurements of some fuel types

Few measurements were made from domestic, commercial and industrial waste, and the emission factors measured in this study were higher than those observed in previous studies. The effect of moisture content on waste burning has been suggested to impact emissions of particulate matter by around an order of magnitude (Jayarathne et al., 2018). Furthermore, only one LPG stove was used to evaluate emissions from this fuel source, with emissions likely to vary by the type of burner used. Future studies should also make more measurements from waste burning to better understand the effect of composition on emissions. Comprehensive measurements should also be made of emissions from combustion of a range of additional crop residues, as these are an important NMVOC source in India (Jain et al., 2014).

4. Evaluation of the impact on ~~ozone~~O_3 and SOA production as well as the toxicity of emissions

Better understanding of the drivers of photochemical ~~ozone~~O_3 and SOA production from burning emissions is required. A large variety of high molecular weight species with likely low volatilities, such as phenolic and furanic compounds, were released from burning. These NMVOCs are expected to have a large influence on subsequent atmospheric chemistry, and a detailed understanding of this chemistry is required to truly assess the impact of biomass burning on air quality.

5. Evaluation of the relative importance of fuel types to air quality in India

Detailed evaluation of fuel use across India is required to evaluate the relative impact of emissions from fuel wood, municipal solid waste, cow dung cakes and LPG. The emission factors measured for cow dung cake and municipal solid waste in this study were much higher than for fuel wood and LPG and indicated that these sources are likely to contribute significantly to ~~the~~ poor air quality ~~observed in India~~.

The comprehensive characterisation of emissions from fuel types in this study should be used to produce spatially disaggregated local emission inventories to provide better inputs into regional policy and global chemical transport models. This should allow a better understanding of the key drivers of poor air quality in India and could allow meaningful mitigation strategies to alleviate the poor air quality observed.

Author contributions. GJS made measurements with GC×GC-FID, combined and analysed datasets and lead the writing of the manuscript. WJFA made measurements of NMVOCs by PTR-ToF-MS, supported by CNH, LKS and NT. BSN made measurements with DC-GC, supported by JRH. ARV assisted in running and organising of experiments. RA, AM, RJ, SA, LY and SKS collected fuels, carried out burning experiments and measured gas volumes up the flue. RED worked on GC×GC-FID method development. SSMY assisted with data interpretation. EN, NM, RG, ARR and JDL worked on logistics and data interpretation. TKM and JFH provided overall guidance with setup, conducting, running~~,~~ and interpreting experiments. All authors contributed to the discussion, writing, and editing of the manuscript.

Competing interests. The authors declare that they have no conflict of interest.

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