METHOD DEVELOPMENT FOR THE CHARACTERIZATION OF MAINSTREAM BIDI SMOKE BY GAS CHROMATOGRAPHY-MASS SPECTROMETRY (GC-MS) AND INFRARED SPECTROMETRY (IR)

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RESEARCH OBJECTIVES FOR BIDI SMOKE ANALYSIS

- To identify and develop effective GC-MS techniques for analyzing the toxic constituents of bidi smoke
- To analyze quantitatively, the constituents of Bidi cigarette by infrared spectroscopy using single puff analysis

REFERENCE ETHYLENE SPECTRA AND BIDIS’ CO SPECTRA OBTAINED AT DIFFERENT FLOWRATES.

GENERAL OVERVIEW OF BIDI CIGARETTES

- Made in India and exported to other countries
- Smoked by tens of millions of Indians
- Available in different brands
- Bidi cigarettes are hand-rolled
- Very few qualitative and quantitative studies have been reported on the Bidi cigarette to date.

BIDI SMOKE SAMPLING FOR FTIR ANALYSIS.

- An evacuated 2.4-meter gas cell was used for the collection of the gaseous portion of the mainstream smoke from a lit bidis.
- The pre-weighted bidis were connected to the sampling bags and then to a 0.5 µm cartridge filter which traps the particulate matter, before the gaseous components get into the gas cell.
- Sampling was done for 6 seconds for each sampling period, for puff by puff analysis.

ANALYSIS OF BIDI SMOKE USING FTIR

- Analysis was done on a Nicolet Magna 550 Fourier Transform Infrared Spectrometer.
- The 2.4-meter gas cell filled with the bidi smoke sample was taken to the FTIR for analysis, with the background spectra of the empty cell already taken before sampling.
- Analysis was carried out at 0.5 cm⁻¹ resolution, 128 scans, Happ-Genzel apodization and no zero-filling.
- Spectra obtained were compared to quantitative standard reference spectra, for identification of constituents.

FTIR ANALYSIS OF BIDIS CIGARETTE SMOKE.

SAMPLING APPROACH FOR GC-MS ANALYSIS

- Sorbent tubes and filter pads were used for sampling bidi smoke analytes for GC-MS analysis
- These sorbent tubes include activated charcoal, CarboxenTM, and Diaionphenylhydrazine (DNPH)
- Analytes adsorbed on the sorbent tubes were extracted using different solvents including methanol, petroleum ether and acetonitrile, for GC-MS analysis optimization.
- Compounds present in the particulate matter on the filter pads were extracted with methylene chloride.

OPERATING CONDITIONS FOR GC-MS

<table>
<thead>
<tr>
<th>Mass Spectrometer</th>
<th>Capillary Temperature</th>
<th>Oven Temperature</th>
<th>Total Chromatographic Run Time</th>
<th>Injector Temperature</th>
<th>Carrier Flow Rate</th>
</tr>
</thead>
<tbody>
<tr>
<td>50-450amu</td>
<td>250°C</td>
<td>320°C</td>
<td>22.5min</td>
<td>260°C</td>
<td>0.9mL/min</td>
</tr>
</tbody>
</table>

CONCENTRATIONS (PPM) OF SELECTED BIDI SMOKE CONSTITUENTS FROM 6 SECOND PUFF BY PUFF FTIR ANALYSIS

<table>
<thead>
<tr>
<th>Compound</th>
<th>Concentration (ppm)</th>
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</thead>
<tbody>
<tr>
<td>CO</td>
<td>12,000</td>
</tr>
<tr>
<td>Ethylene</td>
<td>400 ± 10</td>
</tr>
<tr>
<td>Methanol</td>
<td>520 ± 10</td>
</tr>
<tr>
<td>HCN</td>
<td>780 ± 12</td>
</tr>
<tr>
<td>1,3 Butadiene</td>
<td>80 ± 10</td>
</tr>
</tbody>
</table>

CONCENTRATIONS OF CARBON MONOXIDE IN BIDI SMOKE AS A FUNCTION OF FLOW RATE FOR A 6 SECOND SAMPLING PERIOD.

GC-MS ANALYSIS OF BIDI SMOKE USING FILTER PADS.

CONCENTRATIONS OF ETHYLENE IN BIDI SMOKE AS A FUNCTION OF FLOW RATE FOR A 6 SECOND SAMPLING PERIOD.

COMPARISON OF REFERENCE AND SAMPLE MASS SPECTRA OBTAINED FOR 2,4-BIS (1,1-DIMETHYL-ETHYL PHENOLS) USING CARBOXEN

CONCLUSION

- Detection of carbonyls such as acetaldehyde, propanal was achieved with the use of DNPH sampling media.
- Pyridines, indoles, phenols and steroids were detected in the particulate matter on filter pads.
- Constituents such as nicotine, phenols and hydrocarbons were effectively detected using the activated charcoal sorbent tubes.
- Hydrocarbon compounds with up to 4 carbons or less and inorganic gases such as HCN, CO, and NH₃ can be readily detected with extractive FTIR on a puff-by-puff basis and their concentrations are actually flowrate dependent.

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