The End of Oil Immersion Microscopy?

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EngD Project Background

• MSci Chemistry - University of Nottingham

• October 2015 - EPSRC Centre for Doctoral Training in Carbon Capture and Storage and Cleaner Fossil Energy

• Using image analysis to predict how fuel types might perform in a pulverised fuel boiler. Burnout efficiency, slagging, fouling and NO$_x$ /SO$_x$ emissions.
Image Analysis

- Rapidly characterise fuels to predict boiler performance
- A fully automated tool for plant operators
- Blend or as a single fuel source
- Many behavioural aspects of coals can be ‘seen’ under an oil immersion microscope, there is no current technique for collating this information together.

To develop several new image analysis methods to measure coal, char, mineral and ash materials resulting in a simple method that can characterise fuel in a way that enables power generators to understand the consequences of fuel choices.
Oil Immersion Microscopy
Kinetics Free Combustion Simulations...?
Pyrite Detection
30 x 30 O.I image
Mineral Liberation Analysis
Char ‘Ash’
Minerals vs Macerals with SEM imaging
# Blending Strategy

<table>
<thead>
<tr>
<th>Criterion</th>
<th>Grade</th>
<th>Final Blend</th>
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<tbody>
<tr>
<td>Petrographic Composition</td>
<td>🟢🟢🟢</td>
<td>🟢</td>
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<tr>
<td>Pyrite Characteristics</td>
<td>🔴🟢🟢</td>
<td>🔴</td>
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<tr>
<td>Ash Composition</td>
<td>🟢🟢🟢</td>
<td>🟢</td>
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Oil Immersion Image Analysis

• Image Analysis has helped to improve coal and char assessment significantly over the last 20-30 years

• It remains a challenge to combine all the useful characteristics that are measured using EM/OI

• Predicting all major events (boiler performance, slagging and fouling and EP performance) would be a powerful tool for generators
This Research

• Traditionally – Oil Immersion
• Recently - Air microscopy has been used to carry out coal and mineral analysis
  – ‘Cleaner’
  – Analysing much larger areas
  – Identification of mineral matter
• Air immersion vs oil immersion microscopy with 12 different carbon materials coals, cokes and chars
• 12 different carbon materials were selected to represent a wide reflectance range from low rank coal, high volatile bituminous coals, low volatile bituminous, semi-anthracites through to coke material.
• Resin blocks were prepared and polished for each sample to allow air (at 100x magnification) and oil immersion microscopy (at 500x magnification) analysis.
• Automated petrographic analysis to evaluate macerals, microlithotypes and reflectance.
Air vs Oil Mosaic of Low Rank Russian
Air vs Oil Mosaic of Semi-Anthracite
Air vs Oil Mosaic of Coke
Insert an air and oil mosaic of South African (12)
# 12 samples of coal/coke

<table>
<thead>
<tr>
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</thead>
<tbody>
<tr>
<td>Vitrinite</td>
<td>85.2</td>
<td>88.4</td>
<td>72.0</td>
<td>57.0</td>
<td>49.4</td>
<td>49.6</td>
<td>N/A</td>
<td>97.6</td>
<td>91.2</td>
<td>84.0</td>
<td>84.2</td>
<td>48.0</td>
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<tr>
<td>Liptinite</td>
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<td>0.0</td>
<td>3.2</td>
<td>1.0</td>
<td>6.2</td>
<td>0.0</td>
<td>N/A</td>
<td>0.0</td>
<td>0.4</td>
<td>3.0</td>
<td>0.0</td>
<td>1.0</td>
</tr>
<tr>
<td>Semi-Fusinite</td>
<td>5.4</td>
<td>3.6</td>
<td>9.2</td>
<td>18.0</td>
<td>27.8</td>
<td>39.6</td>
<td>N/A</td>
<td>1.7</td>
<td>4.4</td>
<td>6.0</td>
<td>7.4</td>
<td>23.0</td>
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<tr>
<td>Fusinite</td>
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<td>8.0</td>
<td>15.6</td>
<td>24.0</td>
<td>16.6</td>
<td>10.8</td>
<td>N/A</td>
<td>0.7</td>
<td>4.0</td>
<td>7.0</td>
<td>8.4</td>
<td>28.0</td>
</tr>
</tbody>
</table>

| Vitrinite Reflectance (%) | 0.54 | 0.54 | 0.54 | 0.76 | 0.93 | 2.46 | (7.01) | 2.99 | 0.57 | 0.62 | 2.41 | 0.61 |
Vitrinite Peak Position vs Vitrinite Reflectance (%)
Automated vs Manual Vitrinite Reflectance (Oil)
<table>
<thead>
<tr>
<th></th>
<th>Oil</th>
<th>Air</th>
<th>Manual</th>
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</thead>
<tbody>
<tr>
<td>Vitrinite</td>
<td>47.5</td>
<td>48.7</td>
<td>48.0</td>
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<tr>
<td>Liptinite</td>
<td>1.5</td>
<td>1.1</td>
<td>1.0</td>
</tr>
<tr>
<td>SemiFusinite</td>
<td>24.2</td>
<td>23.9</td>
<td>23.0</td>
</tr>
<tr>
<td>Fusinite</td>
<td>26.8</td>
<td>26.3</td>
<td>28.0</td>
</tr>
</tbody>
</table>

![Graph showing frequency distribution across different grey scales for Oil and Air samples compared to Manual analysis.](image-url)
## Conclusions

<table>
<thead>
<tr>
<th>Advantages of Oil Immersion</th>
<th>Advantages of Air Immersion</th>
</tr>
</thead>
<tbody>
<tr>
<td>Superior contrast</td>
<td>Larger captured area</td>
</tr>
<tr>
<td>Less sensitivity to sample blemishes</td>
<td>‘Cleaner’</td>
</tr>
<tr>
<td></td>
<td>Much greater reflectance range (0.5 – 8.0)</td>
</tr>
</tbody>
</table>
To be Continued...

• The size and shape of each particle
• Characterised using mineral liberation analysis
• Thermal analysis for intrinsic reactivity
• Size & shape using a Malvern Camsizer

• Pyrolysis in a Drop tube furnace at 200ms, 1% oxygen and 1300°C to produce a devolatilised char sample.
• Char samples analysed using air and oil immersion microscopy, thermal analysis, density and particle size and shape – for comparison with the initial samples.
Acknowledgements

• EPSRC EngD Centre
• BF2RA for funding the project