School of Chemical and Process Engineering (SCAPE)
Faculty of Engineering

UNIVERSITY OF LEEDS

Experience and Limitations found with X-ray Fluorescence (XRF) Analysis on Biomass Fuels, Low Grade Coal and Agglomerates [Work in Progress]

Author(s)

Mr Stephen Chilton & Dr William Nimmo

Introduction

UNIVERSITY OF LEEDS

Bubbling fluidised bed combustor

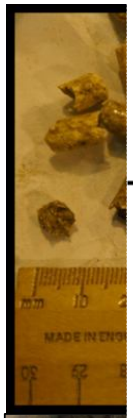
- 350kW thermal
- Silica sand
- 0.5m^2 x 0.2-0.3m bed
- 30-35 Kg/hr fuel flow
- Approx. 9000L/min air
- 800-900°C

Measurements

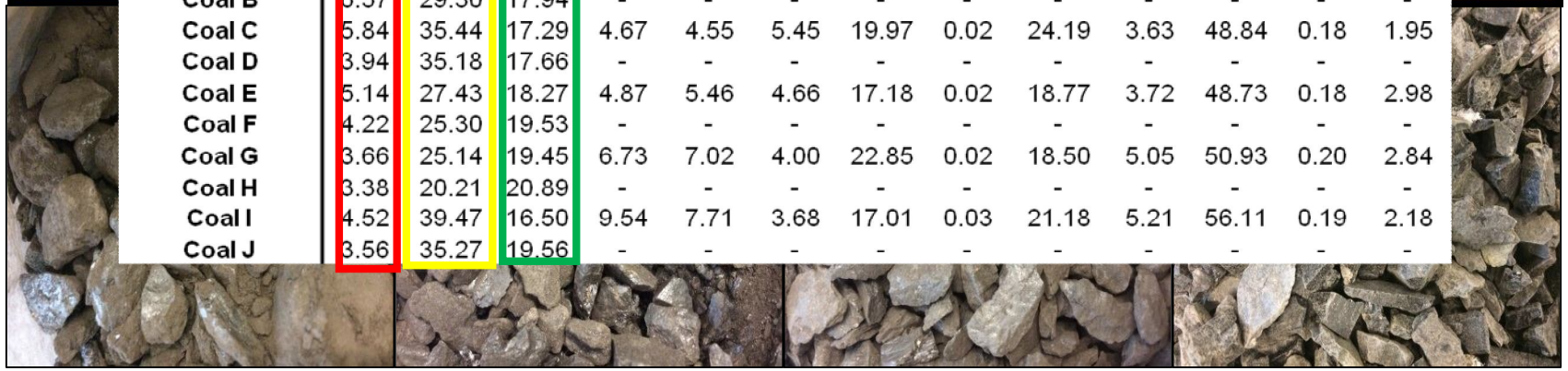
- Analysers [O_2/NO_x/SO_2/CO/CO_2]
- Labview
  - Pressure
  - Temperature
- Fly ash
- Bottom ash
- Bed
- Agglomerates

Low grade Fuel(s)

UNIVERSITY OF LEEDS



	S (wt%)	Ash (wt%)	HHV (MJ/Kg)	Sodium %	Magnesium %	potassium %	iron %	manganese %	aluminium %	Calcium %	Silicon %	Phosphorus %	Titanium %
Peanut	<0.1	4.04	19.14	21.58	5.62	2.12	3.17	0.24	9.07	8.36	44.01	4.87	1.21
Wheat Straw	<0.1	8.26	15.69	9.61	6.31	6.61	4.34	1.69	7.48	22.69	39.77	2.24	0.46
Miscanthus	<0.1	4.76	17.15	16.18	4.77	1.65	0.90	0.17	0.80	3.92	55.98	5.25	0.05
Wood	<0.1	1.14	17.65	11.65	3.24	2.95	2.60	0.14	1.42	13.52	56.54	3.85	0.07
Oat	<0.1	4.23	17.34	19.76	3.60	6.26	1.04	0.06	1.58	21.14	38.38	4.76	0.26
Coal A	6.73	39.91	16.60	3.79	4.79	4.89	22.62	0.02	26.70	3.01	51.92	0.18	2.61
Coal B	6.57	29.30	17.94	-	-	-	-	-	-	-	-	-	-
Coal C	5.84	35.44	17.29	4.67	4.55	5.45	19.97	0.02	24.19	3.63	48.84	0.18	1.95
Coal D	3.94	35.18	17.66	-	-	-	-	-	-	-	-	-	-
Coal E	5.14	27.43	18.27	4.87	5.46	4.66	17.18	0.02	18.77	3.72	48.73	0.18	2.98
Coal F	4.22	25.30	19.53	-	-	-	-	-	-	-	-	-	-
Coal G	3.66	25.14	19.45	6.73	7.02	4.00	22.85	0.02	18.50	5.05	50.93	0.20	2.84
Coal H	3.38	20.21	20.89	-	-	-	-	-	-	-	-	-	-
Coal I	4.52	39.47	16.50	9.54	7.71	3.68	17.01	0.03	21.18	5.21	56.11	0.19	2.18
Coal J	3.56	35.27	19.56	-	-	-	-	-	-	-	-	-	-



Options

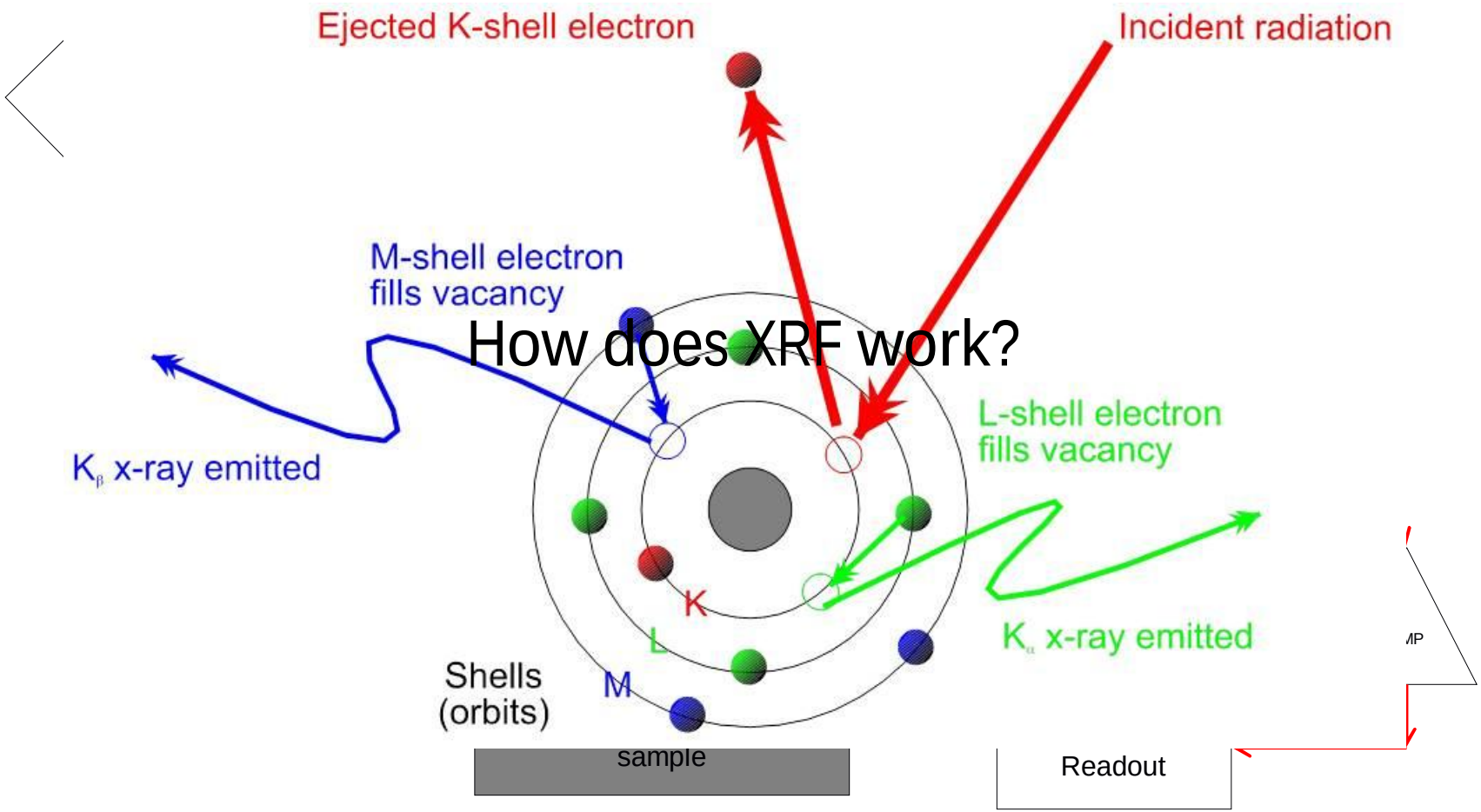
UNIVERSITY OF LEEDS

- How to analyse slag, agglomerates, fouling samples?
  - Optical Emission spectrometry (OES)
  - Atomic Absorption Spectrometry (AAS)
  - Inductively Coupled Plasma (ICP-OES)
  - Wet chemistry

- Factors of choice:
  - Time
  - cost
  - Complexity
  - Training
  - Accuracy
  - Repeatability
  - Portability
  - Result?

X-ray fluorescence (XRF)

UNIVERSITY OF LEEDS



Investigation & scope

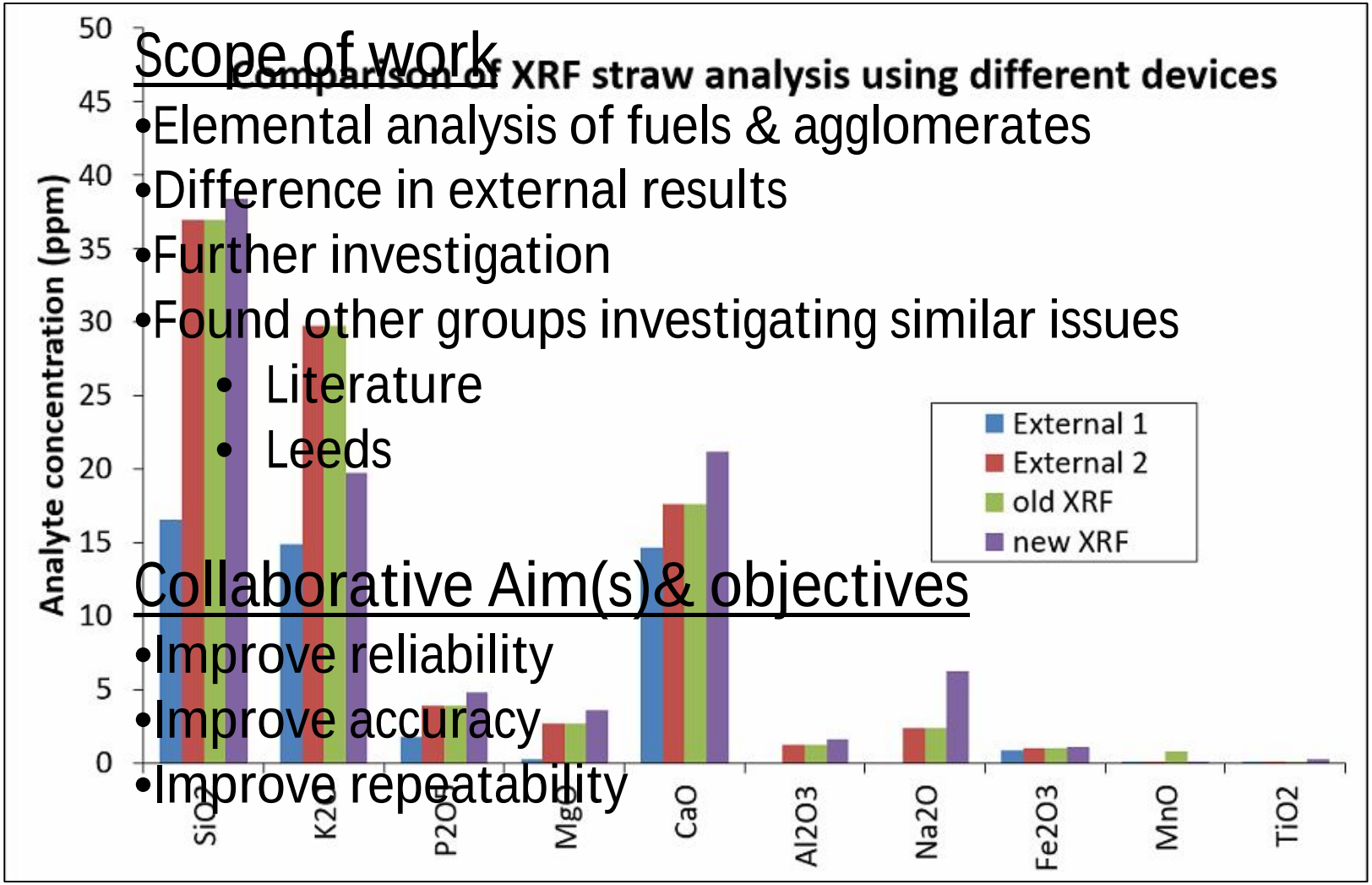
UNIVERSITY OF LEEDS

Scope of work

- Elemental analysis of fuels & agglomerates
- Difference in external results
- Further investigation
- Found other groups investigating similar issues
  - Literature
  - Leeds

Collaborative Aim(s)& objectives

- Improve reliability
- Improve accuracy
- Improve repeatability

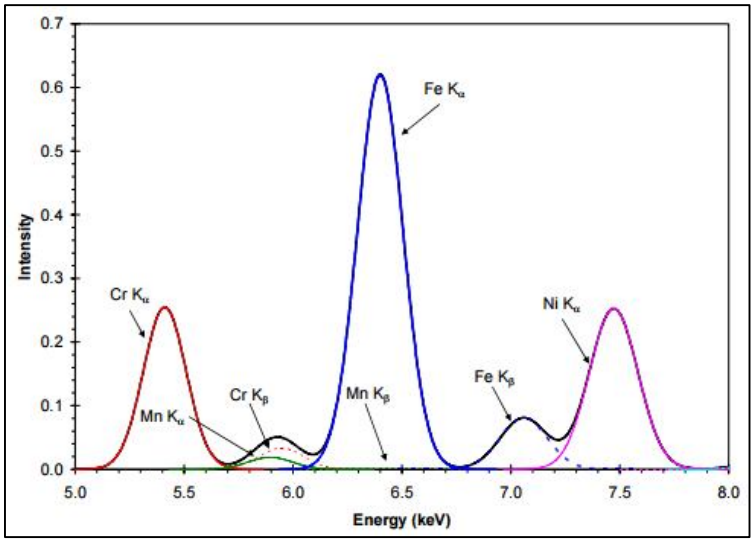


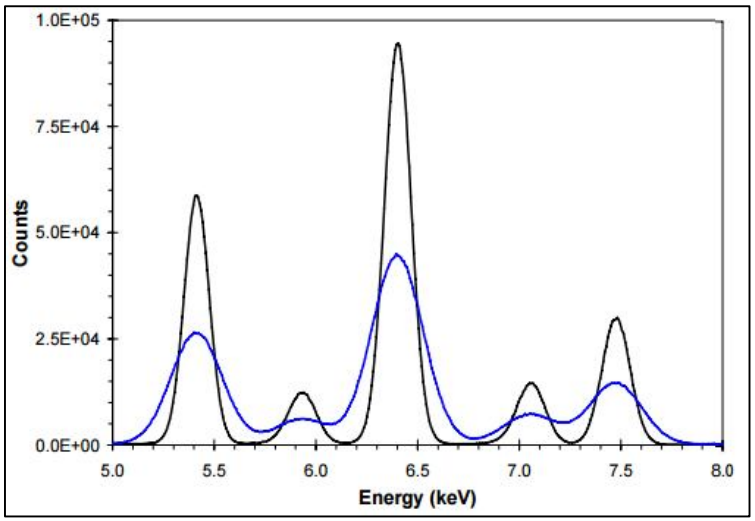
XRF limiting factors

UNIVERSITY OF LEEDS

Factors effecting XRF results

- User ability/training
- Type of analysis
  - Quantitative
  - Semi-Quantitative
  - Qualitative
- Power/size of device
- XRF Software/algorithms
  - Standard-less fundamental parameters (SLFP)
  - normalisation
- Sample preparation
- Sample chemistry/physiology
- Methodology





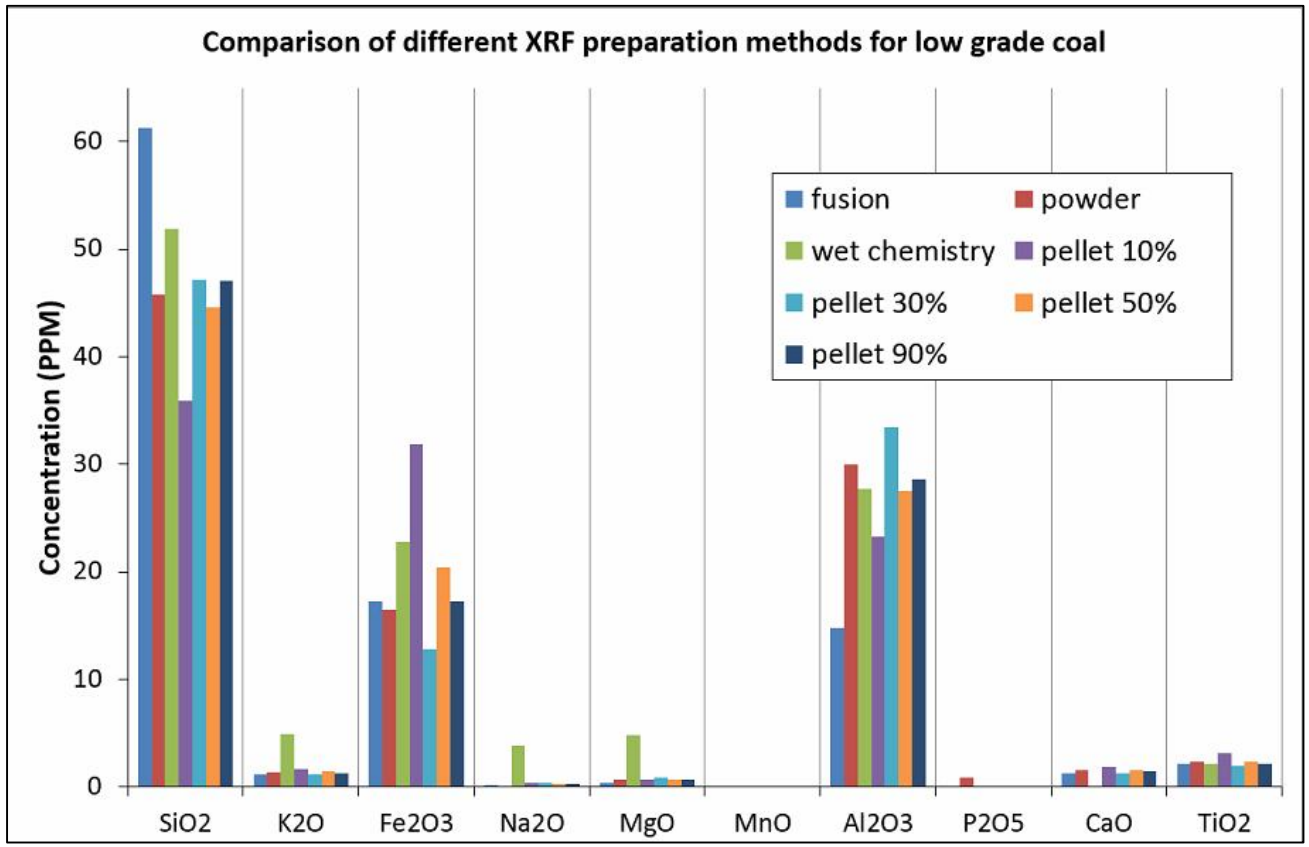
[Amp-

Sample preparation

UNIVERSITY OF LEEDS

Different methods
- Fusions
- Powders
- Push pellets

Factors
- Particle size
- Optical distance
- Bulk density
- X-ray penetration
- Homogeneity



Ashing

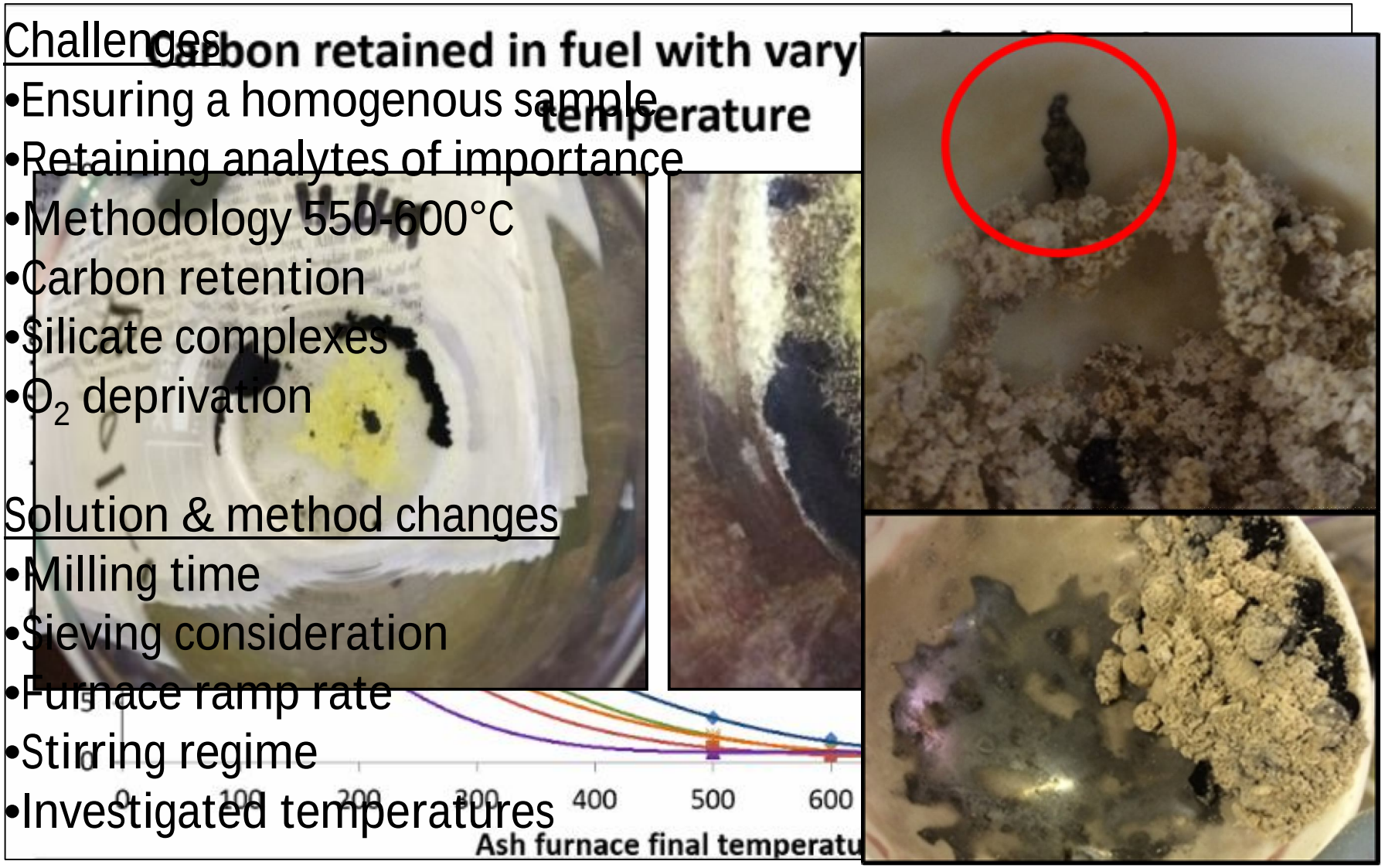
UNIVERSITY OF LEEDS

Challenges

- Ensuring a homogenous sample
- Retaining analytes of importance
- Methodology 550-600°C
- Carbon retention
- Silicate complexes
- O_2 deprivation

Solution & method changes

- Milling time
- Sieving consideration
- Furnace ramp rate
- Stirring regime
- Investigated temperatures



Method development

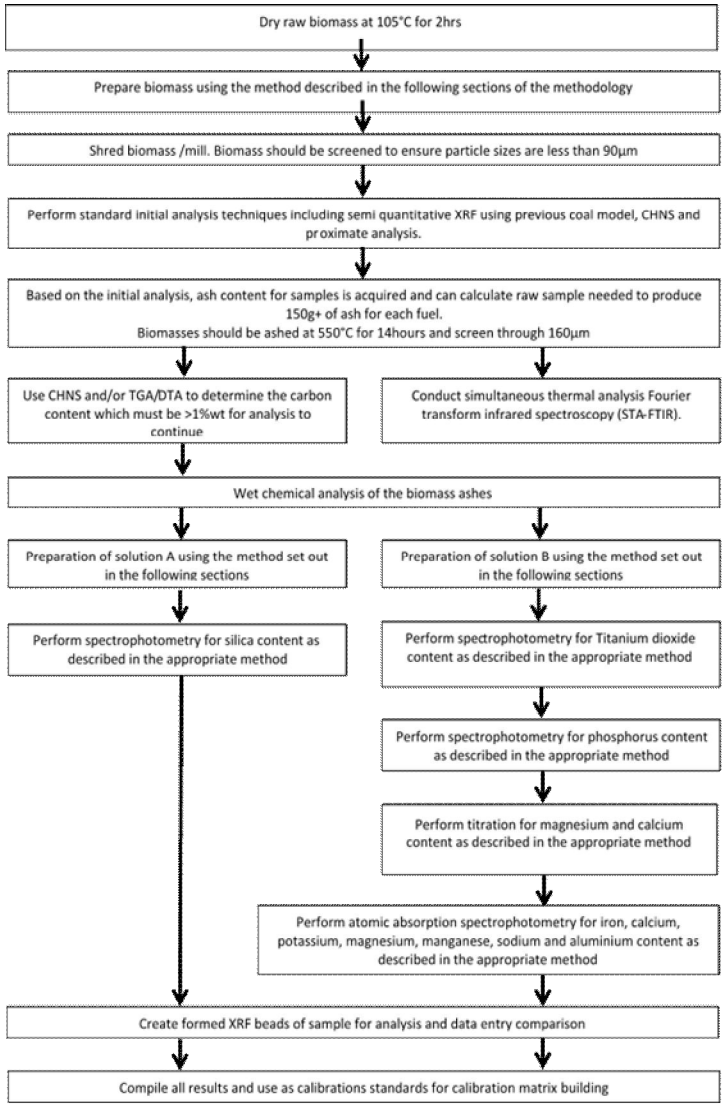
UNIVERSITY OF LEEDS

Investigated

- Carbon content
- Particle size
- Wet chemistry method
- Ashing method
- Device variance
- Program parameters
- Making own standards

Method development

- Systematic method
- Work with other groups
- Reduce result differences



Conclusions

UNIVERSITY OF LEEDS

- Use a standardised method for comparison
- Use results with caution
- Reduced errors in repetition
- Work in progress
- Larger community addressing problems
- Can XRF be used for low grade fuels and agglomerates?

Sample preparation	(Loubser and Verryn, 2008, Anzelmo, 2009, Gazulla et al., 2009, Stankova et al., 2010, Wang et al., 2010, Gazulla et al., 2010, Matsunami et al., 2010, Pease, 2013, Le Roux and De Vleeschouwer, 2010, LUO et al., 2011)
XRF analysis technique(s) & methodology development	(Gazulla et al., 2010, Fernández-Ruiz et al., 2010, Robinson et al., 2009, Andersen et al., 2013, Morgan et al., 2015, Teng et al., 2013, de Jonge and Vogt, 2010, Terzano et al., 2013)
Review(s)	(Evans et al., 2014, Taylor et al., 2014, Clough et al., 2014, Gibson et al., 2014, Butler et al., 2015)

Thank you

Any questions?