LEHIGH UNIVERSITY

Integration of LIBS with Machine Learning for Real-Time Monitoring of Feedstock in H_2 Gasification Applications

2023 FECM Spring R&D Project Review Meeting

Project DE-FE0032177

(10/01/2022 – 09/30/2024)

April 18, 2023

ERCo ENERGY RESEARCH COMPANY   GTI ENERGY   SpG Consulting, LLC   ENERGY RESEARCH CENTER

Presentation Outline

- ❑ Project Background
- ❑ Proposed Technology
- ❑ Project Objectives
- ❑ Technical Approach
- ❑ Activities Up to Date

Project Background

- Gasification technologies for non-recyclable plastics, biomass and legacy coal waste are of merit.
- It builds upon technologies developed for coal gasification and favors a future hydrogen circular economy concept.
- DOE has identified the heterogeneity of these waste streams as a significant barrier for any conversion process to be economically viable.
- DOE has included feedstock characterization methods in the list of R&D opportunities to improve:
  - Cost-effectiveness of waste conversion technologies,
  - Process and quality control,
  - Relating feedstock composition to conversion performance.

Project Background

❑ Current State of Technology (SOT)

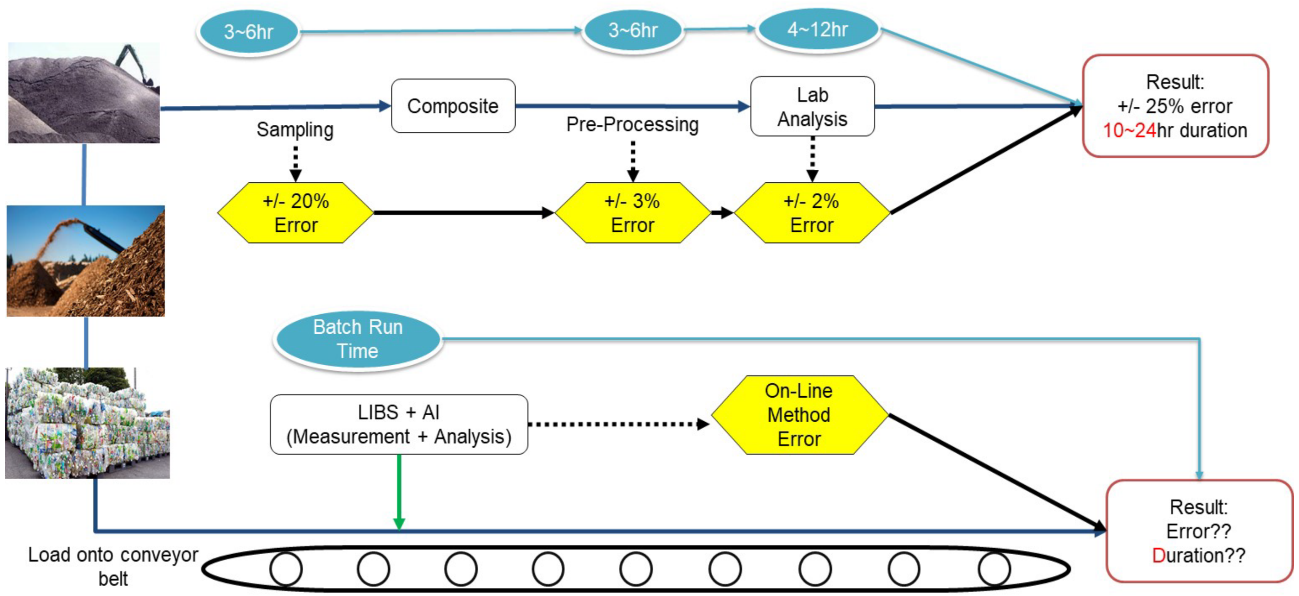
- ❑ Municipal Solid Waste (MSW) is a very heterogeneous material, which makes it difficult for sampling and analysis.
- ❑ No ready-to-use, rapid/real-time measurement techniques available for waste feedstock applications.
- ❑ Critical review of real-time methods for MSW quality monitoring (C. Vrancken et al., 2017):
  - Most techniques can only measure a limited number of parameters
  - **Technologies need additional development to fully adapt them to the gasification environment**
  - **New algorithms are needed for accurate measurements and automatic material identification**
- ❑ Waste feedstock processing presents similarities to other industries, which could improve material management by using advanced analytical technology.

Value Proposition

- Enable continuous on-line measurement of waste feedstock properties to confirm product specifications:
  - Substantially eliminate cost of sampling, compositing and laboratory analysis of feedstock.
  - Improved feedstock processing system performance and product QA/QC
  - Reduced operator interaction with the equipment and waste – improved plant safety

- Feed-forward process control of downstream gasification conversion system
  - Continuous, on-line data on feedstock properties will enable optimization of conversion process operating conditions, including gasification temperature, and steam and air or oxygen feed rates:
    - Increased product yield and revenues
    - Improved product quality
    - Reduced reactant consumption of steam and oxygen
    - Improved process reliability – reduced outage and maintenance costs

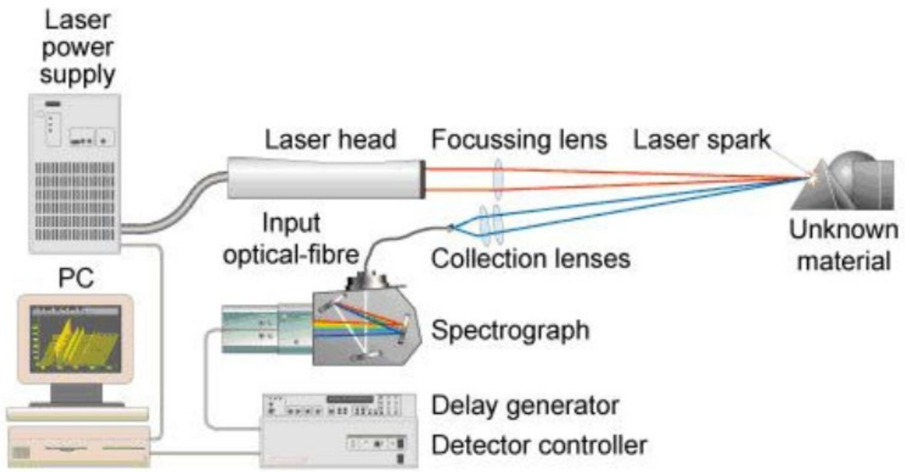
- Increased revenue and reduced O&M costs should yield less than 1 year payback for projected instrument cost in range of $300 - $500K.

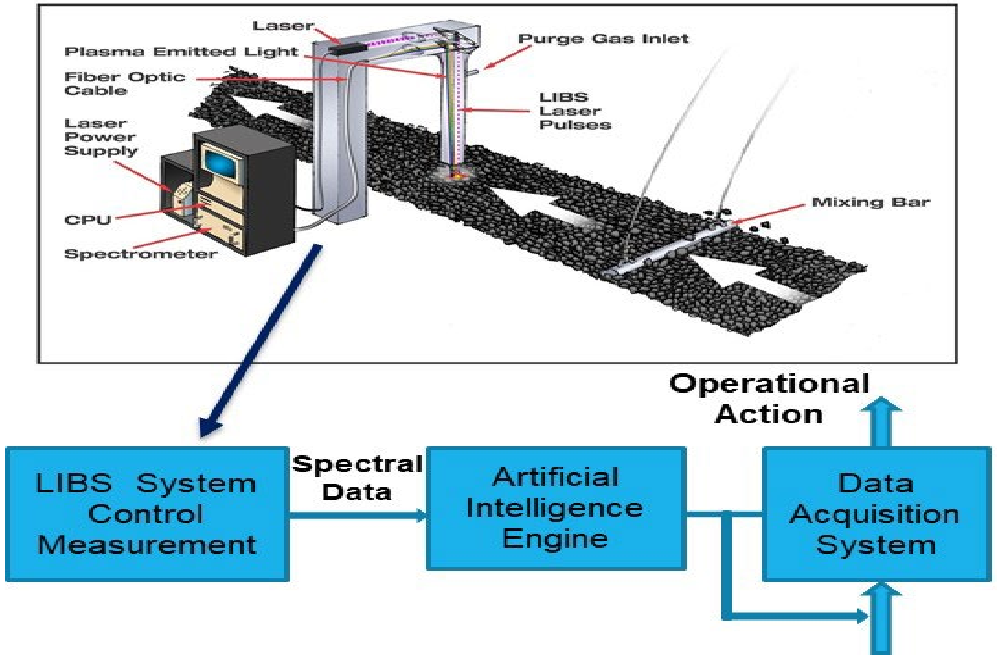


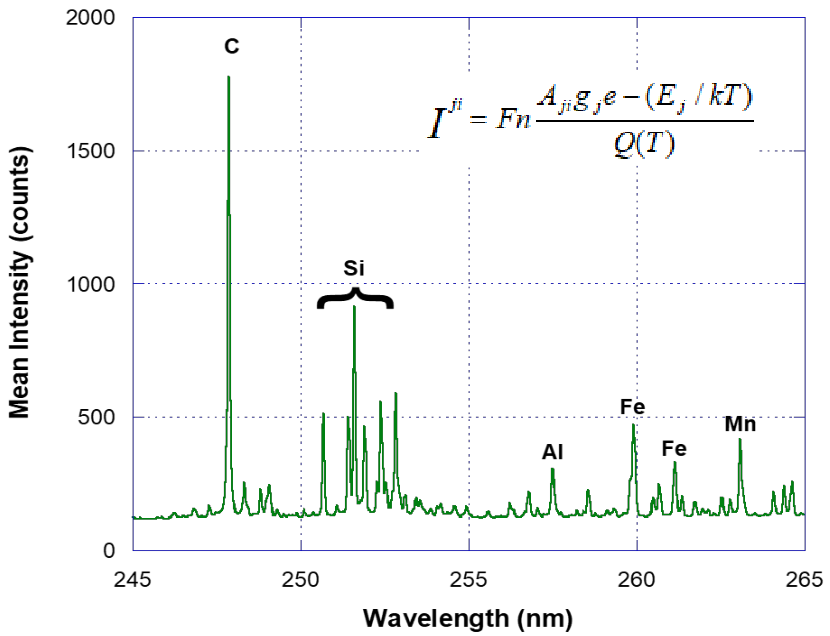
On-Line System Assessment, Based on ASTM D6543

Proposed Technology

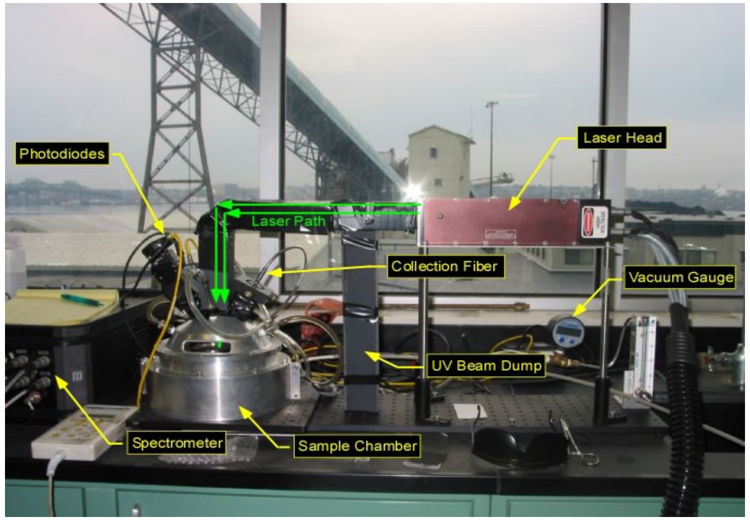
- A system that will allow rapid, in-situ characterization of gasifier feedstock, providing critical data in minutes for continuous confirmation of feedstock specifications, and potential feed-forward process control of downstream hydrogen production.
- This would represent a hundred-fold improvement in the feedstock characterization throughput, over current methods of grab sampling, compositing, and costly laboratory analyses.

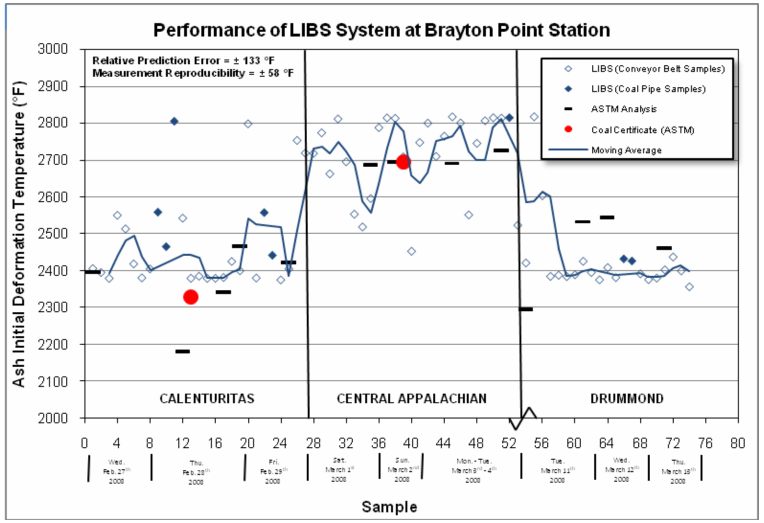


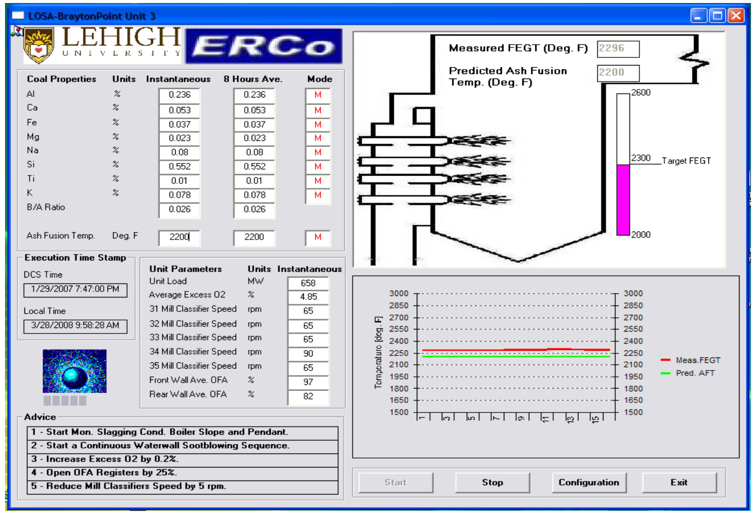




Background Results of Technology







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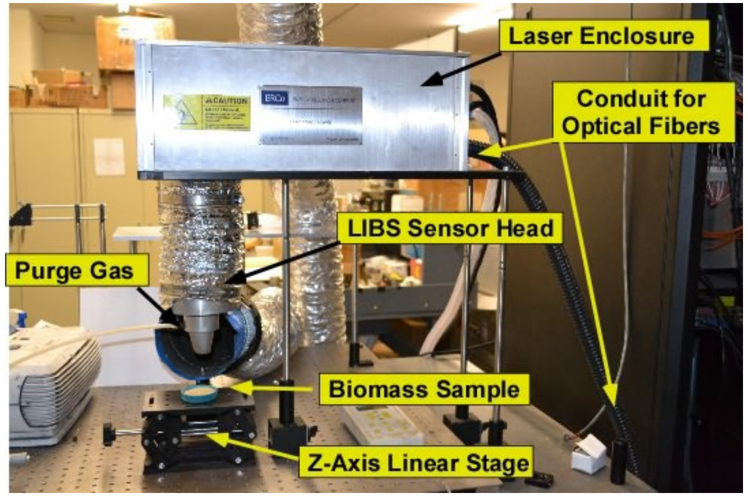

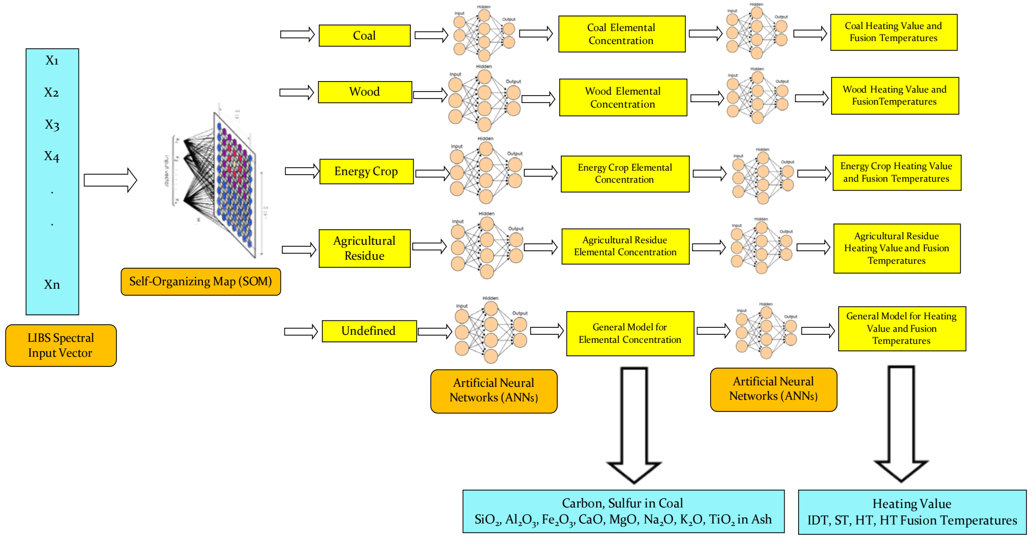

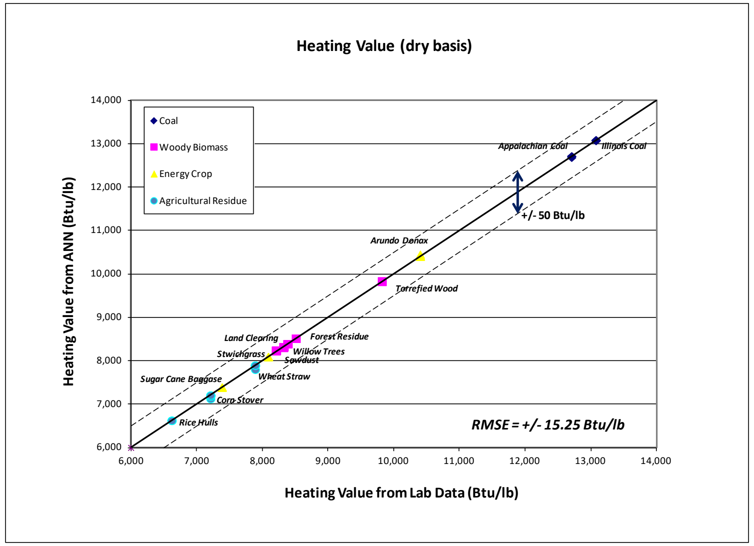

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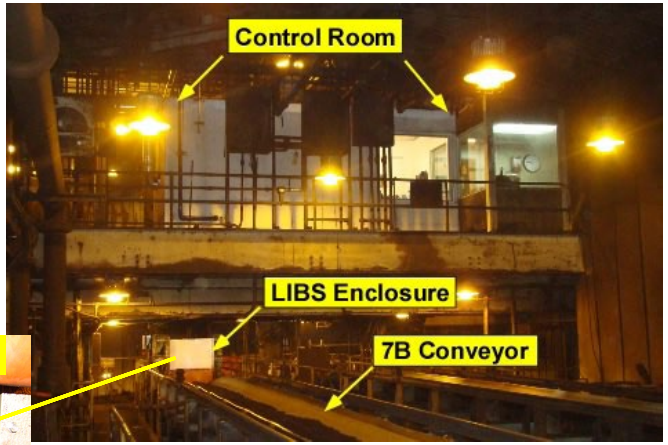

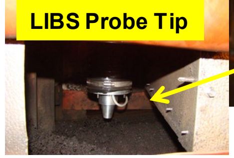

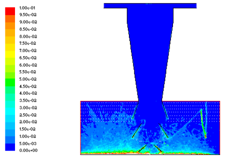

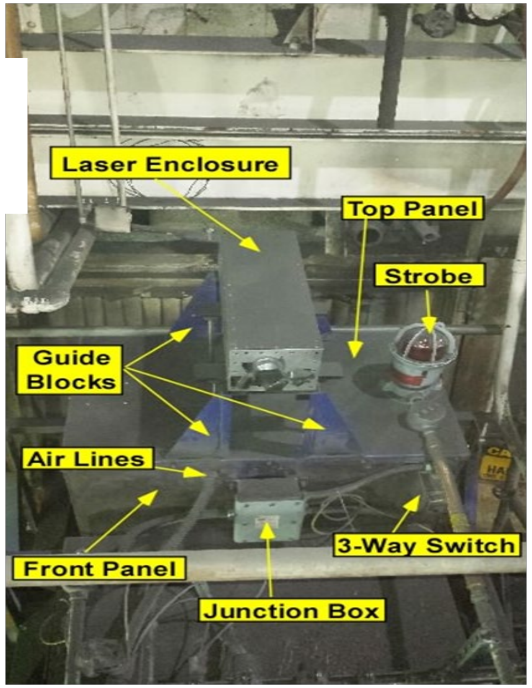

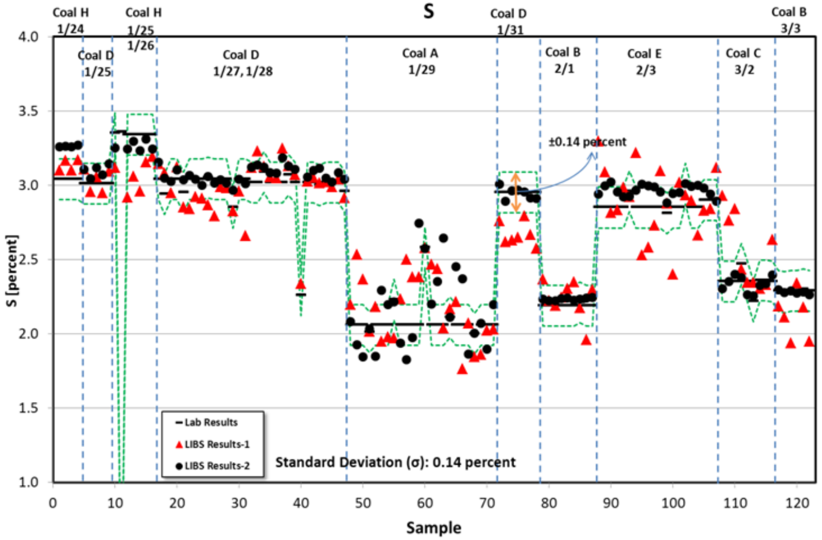

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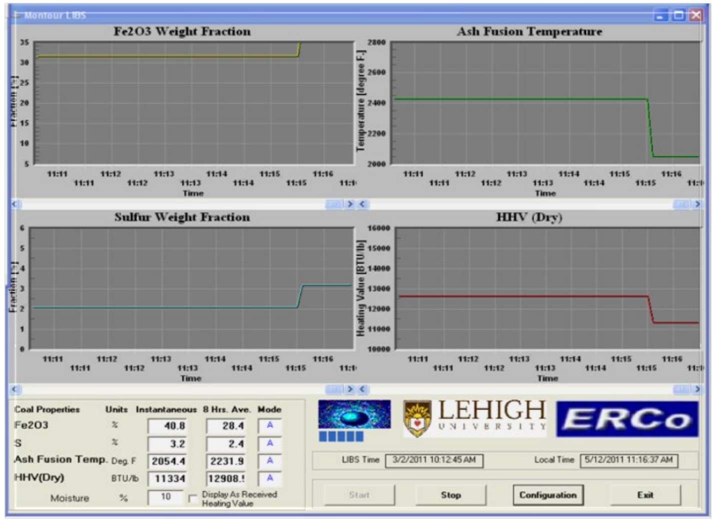

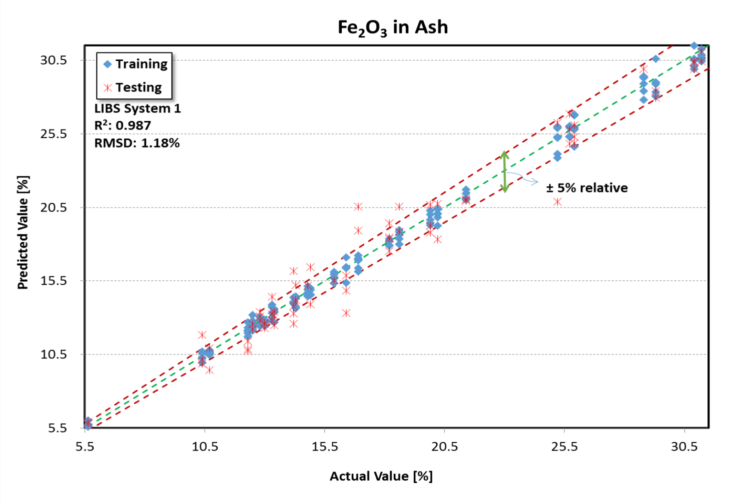

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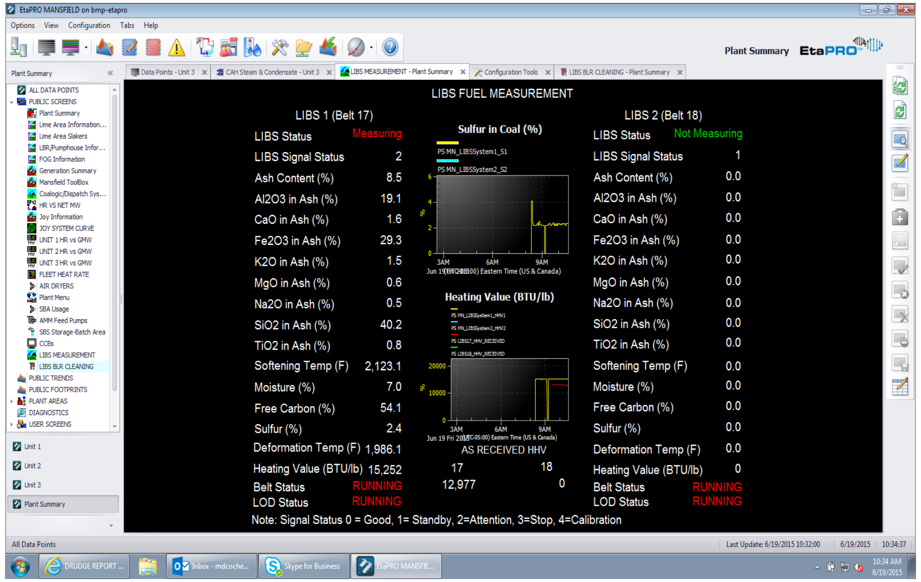
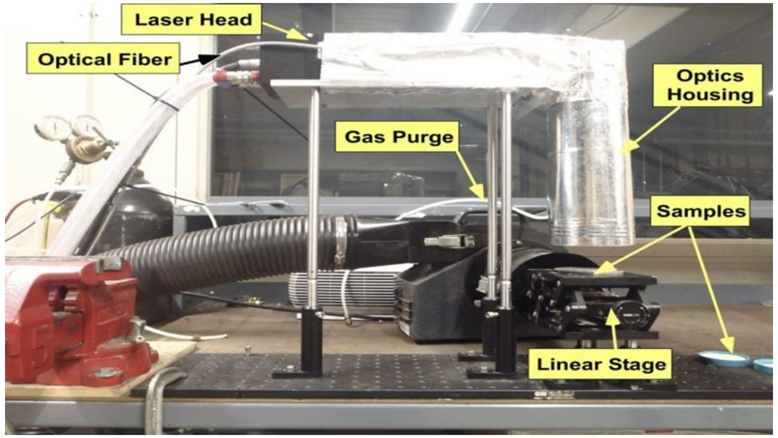
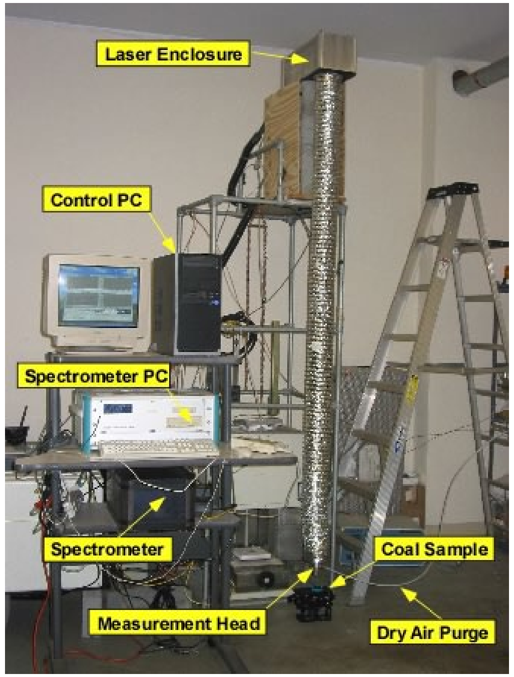


Background Results of Technology





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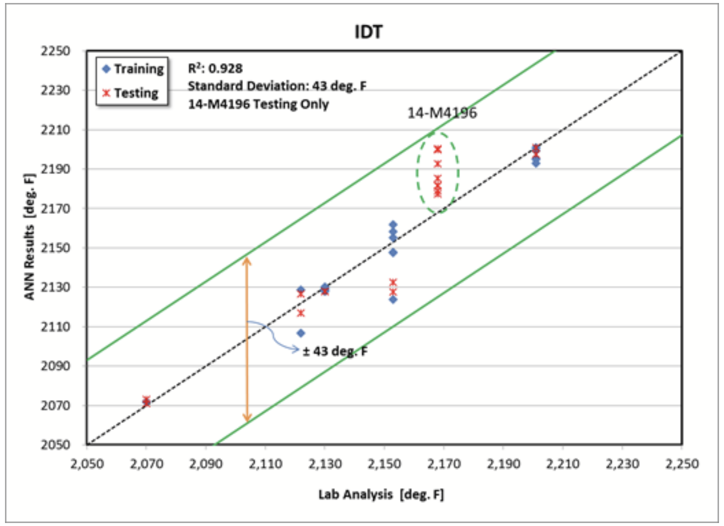

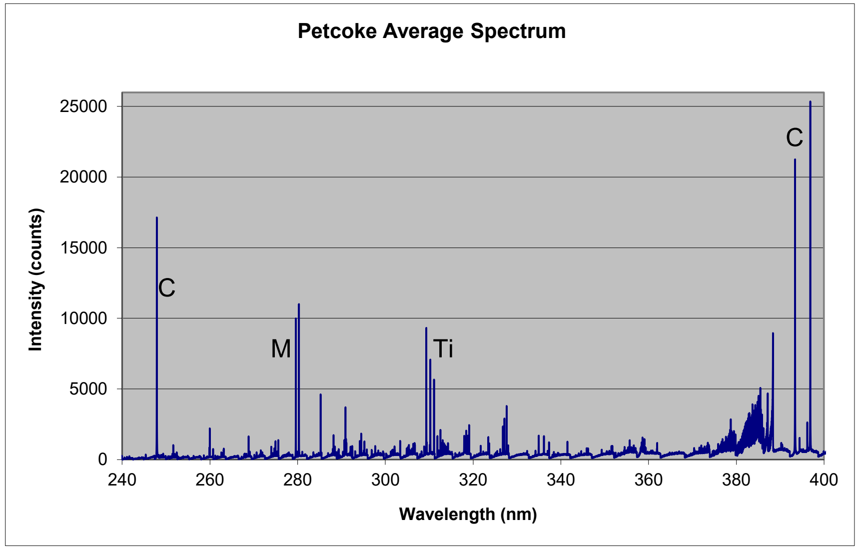

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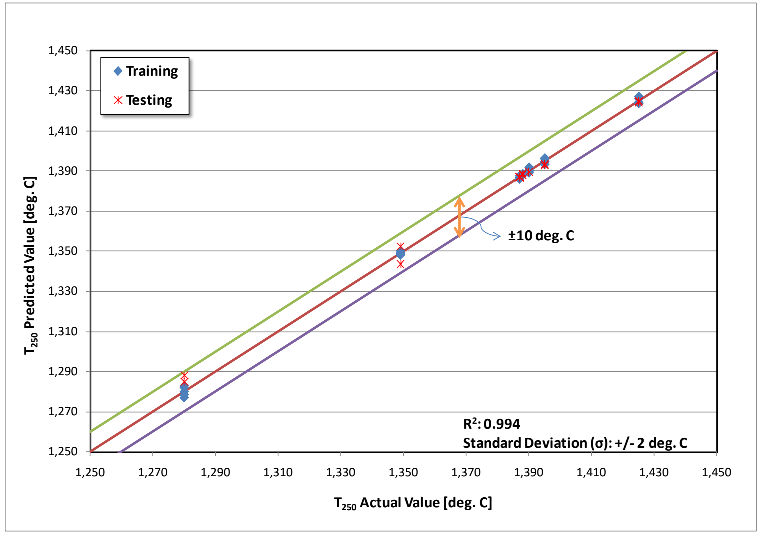


Project Objectives

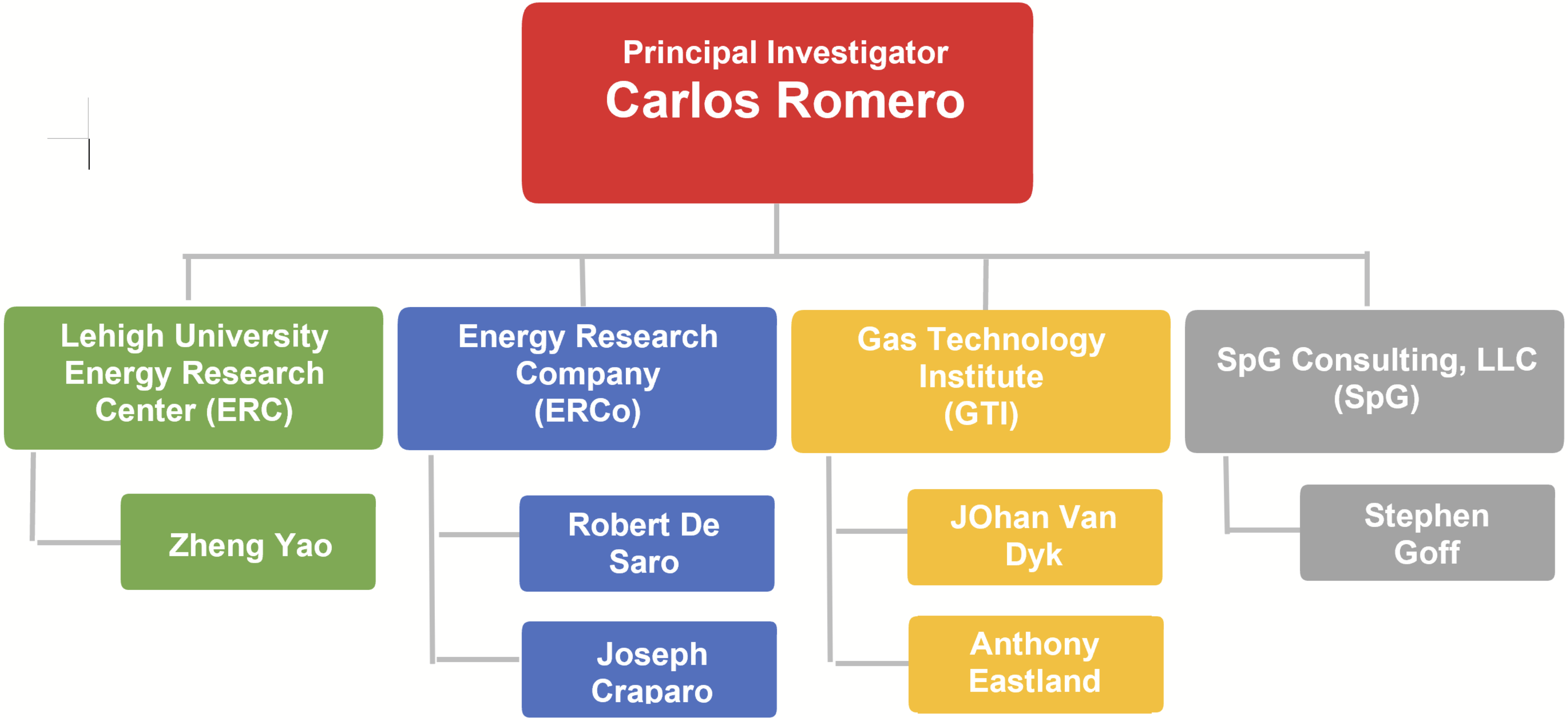
❑ Project Overall Goal

- ❑ Assess the feasibility and potential of using Laser Induced Breakdown Spectroscopy (LIBS) linked to Artificial Intelligence-Machine Learning (AI-ML), for real-time and in-situ chemical analysis of waste materials (biomass, waste plastics and legacy coal waste; individually and in blends), of interest to hydrogen production gasifier operators.

❑ Project Objectives

1. Assemble and analyze a material inventory.
2. Design and assemble a laboratory LIBS system. Optimize measurement technique and develop an analytical database.
3. Develop and validate ML algorithms for LIBS data processing.
4. Assess the benefit of incorporating the proposed system on upgraded operational protocols and control schemes of gasifiers for hydrogen production.
5. Perform a techno-economic analysis (TEA) of the proposed technology integrated with hydrogen gasifiers.

Project Organization



Technical Approach

❑ **Task 1 – Project Management**

1. Project Management Plan (PMP)
2. Technology Maturation Plan (TMP)
3. Environmental Justice Questionnaire (EJQ)

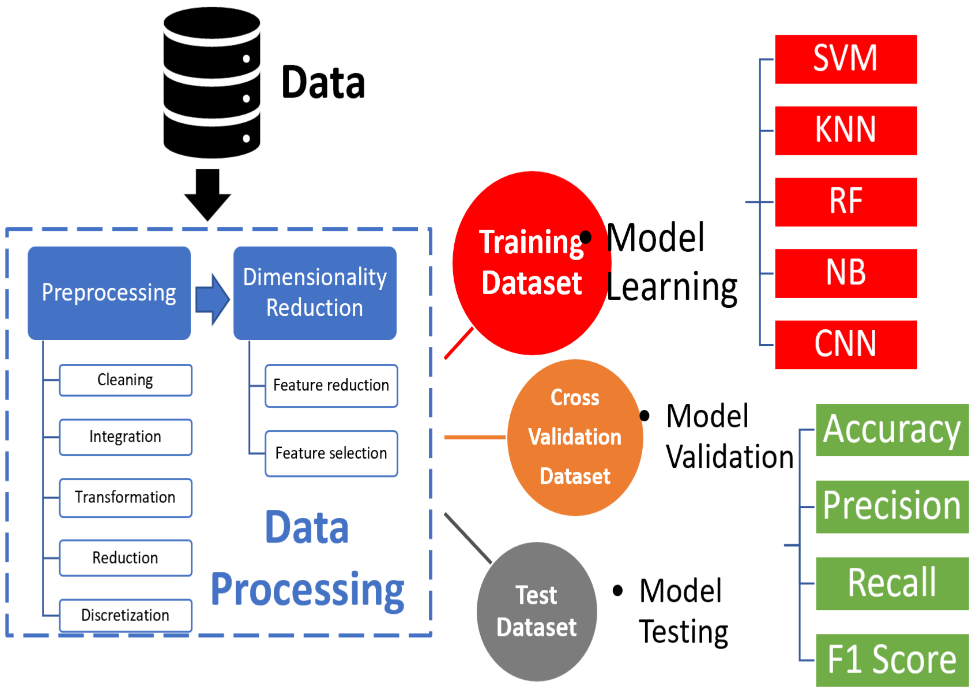
Component	Percentage	Heat Content (dry basis) (Btu/lb)
#1 Polyethylene terephthalate (PET)	40.0	10,250
#2 High density polyethylene (HDPE)	18.0	19,000
#3 Polyvinyl chloride (PVC)	5.9	8,250
#4 Low density polyethylene (LDPE)	18.0	12,050
#5 Polypropylene (PP)	2.0	19,000
#6 Polystyrene (PS)	12.0	17,800
#7 Other*	4.1	13,332
Average Mixed Waste Plastic	100	13,240

❑ **Task 2 – Material Inventory**

1. Material Sampling
   - ❑ Mixed waste plastics, biomass (non-torrefied southern pine, torrefied southern pine, switchgrass) , and legacy coal wastes (Illinois No. 6 bituminous, Montana Rosebud PRB sub-Bituminous, North Dakota lignite).
2. Material Processing and Lab Analysis
   - ❑ Develop a procedure for sample processing, analysis, chain of custody and quality assurance.

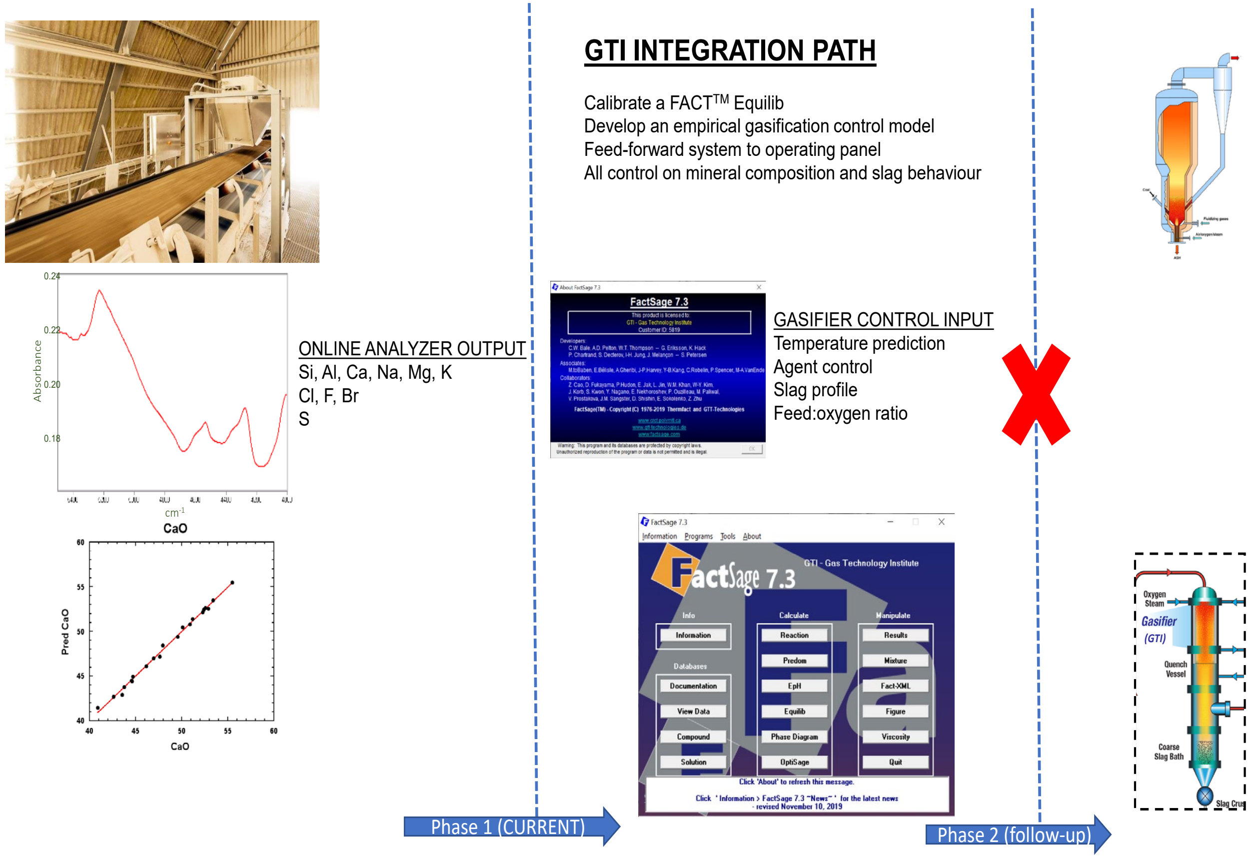
Technical Approach

- Task 3 – LIBS Lab Testing
  - Design and assemble a safe and reliable LIBS system for detection and quantification of material samples under both static and dynamic conditions (material flow on a small-scale research conveyor belt).
  - Optimize measurement technique
  - Develop an analytical database using individual parent sources and blends of all three types of materials.
  - Assess signal-to-noise, R^2 and precision of the LIBS technique
- Task 4 – AI Modeling and Validation
  - Process LIBS data by ML modeling using neural networks, random forest and support vector machine.
  - Parameters of interest: proximate analysis (fixed carbon, ash content, volatile matter, and moisture), ultimate analysis, calorific value, ash composition, chloride content, fusion temperatures (initial and softening), viscosity temperatures (T_{250} and $T_{10,000}$), and thermal conductivity.

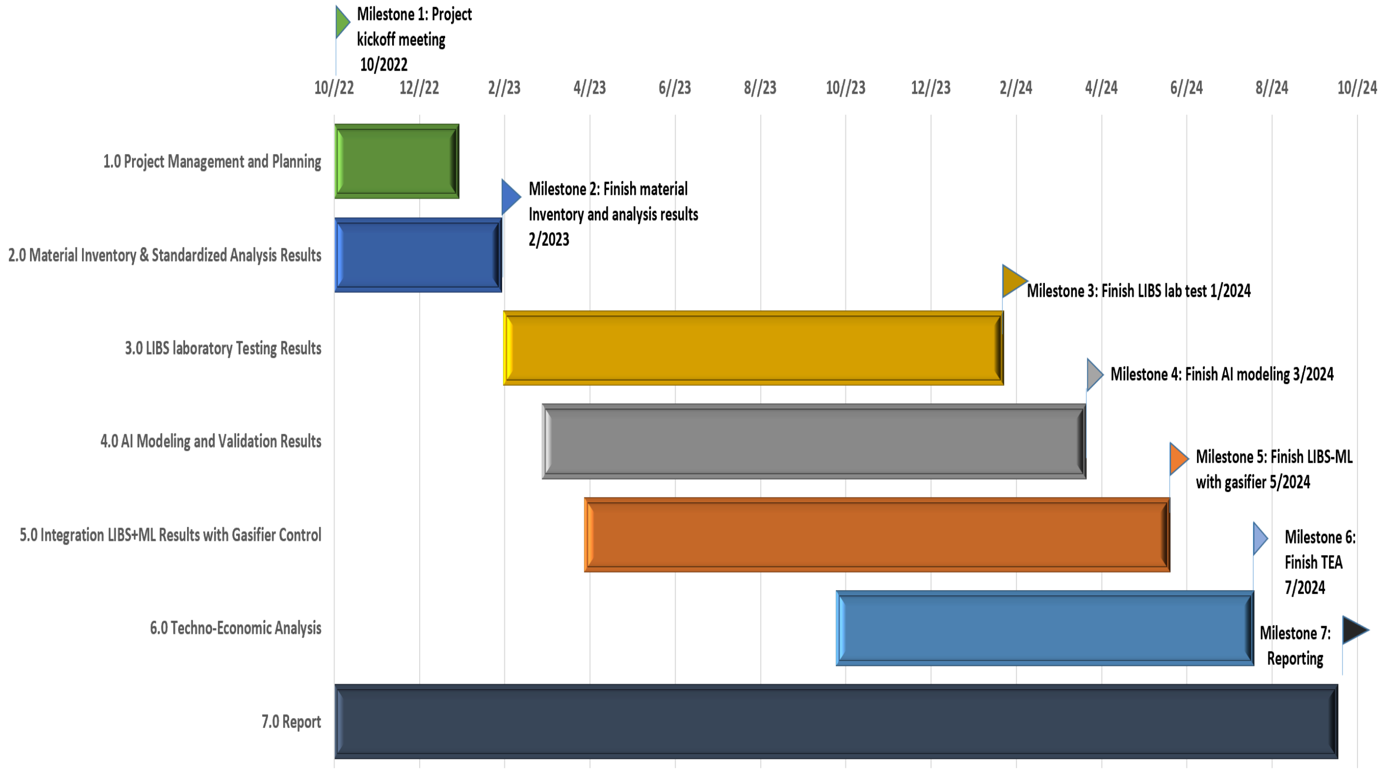


Technical Approach

- Task 5 – Integration of LIBS+ML Results with Gasifier Control
  - Develop phase equilibrium model to predict gasifier operational parameters (i.e., slag profile).
  - Combine empirical gasifier model with phase equilibrium results to provide input/output to a feed-forward gasifier control scheme.
- Task 6 – Technoeconomic Analysis
  - Estimate costs of full-scale system design, procurement and deployment of LIBS+ML system.
  - Perform TEA analysis for the proposed technology integrated with hydrogen gasifiers.



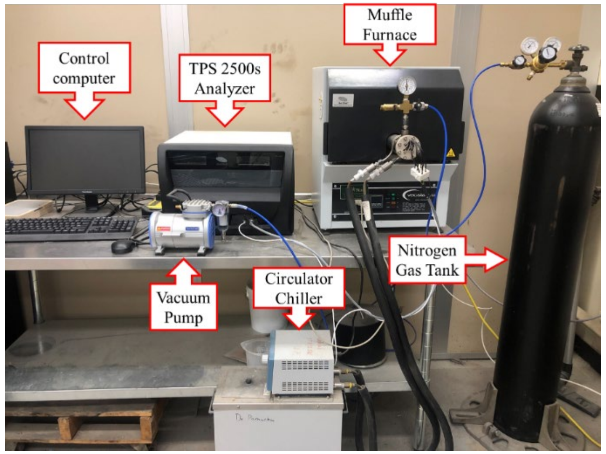
Project Schedule



Task/Subtask Number	Deliverable Title	Due Date
1.1	Project Management Plan (PMP)	Update due 30 days after award. Revisions to the PMP shall be submitted as requested by the NETL Program Manager.
1.2	Technology Maturation Plan (TMP)	Preliminary TMP is due 90 days after award. Updates to the TMP shall be submitted, as needed, throughout the project period of performance. A final TMP is due within 90 days prior to project completion.
1.3	Environmental Justice Questionnaire	Updated Environmental Justice Questionnaire to be submitted as an attachment to the final report.
2.0	Material Inventory List and Standardized Analysis Results	4 months after project start date
3.0	LIBS laboratory Testing Results	16 months after project start date
4.0	AI Modeling and Validation Results	18 months after project start date
5.0	Integration of LIBS+ML Results with Gasifier Control	20 months after project start date
6.0	Techno-Economic Analysis	22 months after project start date
7.0	Report	Periodical and final reports will be submitted according to DOE requirement.

Work on Task 2

- Gathered coal material samples and analysis from Penn State University
- Gathered selected biomass material samples from Idaho National Lab, including torrefied pine.
- Gathered coal refuse and biomass samples from Olympus Power
- Chemical Analyses performed by G&C Analytical Lab
- Thermal conductivity analyses performed at Lehigh University (Hot Disk TPS 2500s Thermal Constant Analyzer)



Idaho National Laboratory

Switchgrass

REFERENCE MATERIAL

Pedigree

Institution: Oklahoma State University
Location: Garvin County, OK
Cultivar: Alamo

Harvested: 2012
Received at INL: 2013
Sample Preparation: Ground to pass through a 1-inch sieve using a Vermeer BG480 grinder

Composition

Table 1. *Chemical composition[a] of Reference Switchgrass (mean of analyses completed 11/2014 & 2/2015)*

%Structural Ash	%Extractable Inorganics	%Structural Protein	%Extractable Protein	%Water Extracted Glucan[b]
1.88	2.07	1.51	0.54	2.28
%Water Extracted Xylan[b]	**%Water Extractives Others**	**%EtOH Extractives**	**%Lignin**	**%Glucan**
0.09	6.68	2.68	16.24	33.21
%Xylan	**%Galactan**	**%Arabinan[c]**	**%Acetate**	**%Total**
21.65	1.43	3.27	3.07	96.60

[a]Determined using NREL "Summative Mass Closure" LAP (NREL/TP-510-48087)
[b]Determined by HPLC following an acid hydrolysis of the water extractives
[c]%Arabinan value includes %mannan, because arabinose and mannose co-elute on the HPLC column

Proximate, Ultimate & Calorimetry

Table 2. *Proximate, ultimate, and calorific values for Reference Switchgrass (reported on a dry basis; completed 6/2014)*

Proximate[a]			Ultimate[b]			Calorimetry[c]	
%Volatile	%Ash	%Fixed Carbon	%Hydrogen	%Carbon	%Nitrogen	HHV	LHV
80.2	4.2	15.6	5.7	47.2	0.5	8077	6749

[a]Proximate analysis was done according to ASTM D 5142-09
[b]Ultimate analysis was conducted using a modified ASTM D5373-10 method (Flour and Plant Tissue Method) that uses a slightly different burn profile
[c]Heating values (HHV, LHV) were determined with a calorimeter using ASTM D5865-10

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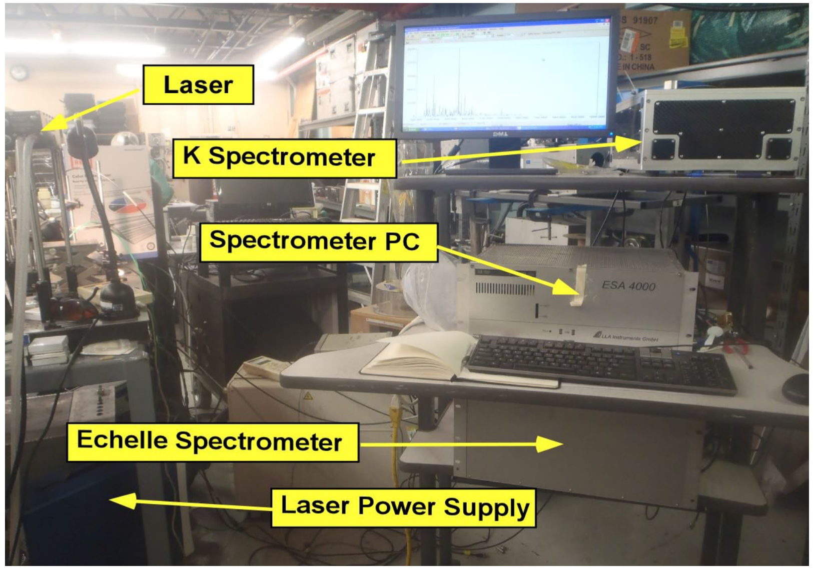
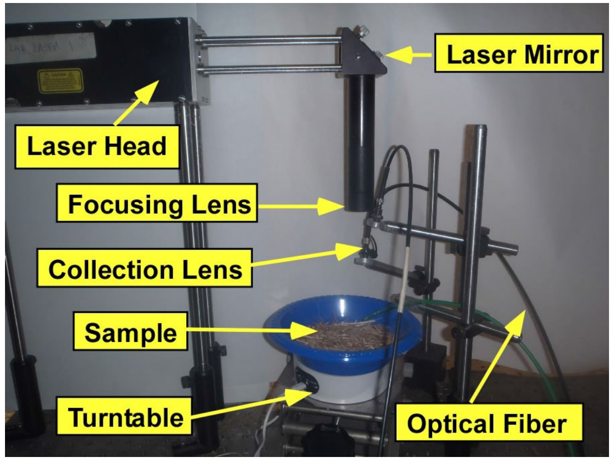
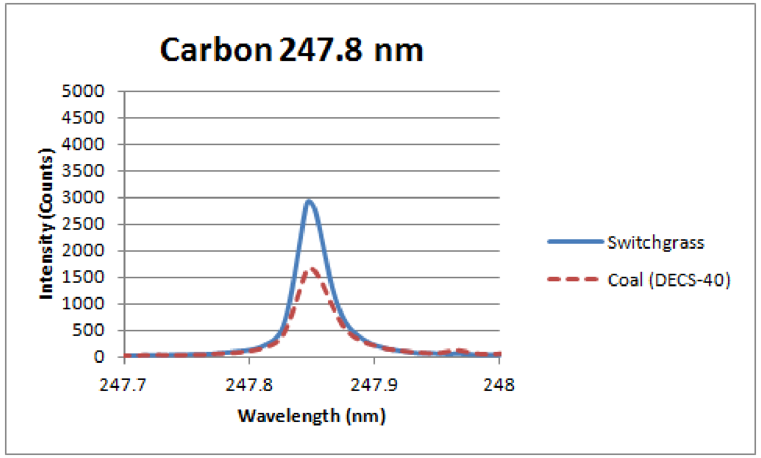
Work on Task 3

- ❑ Completed LIBS laboratory setup
- ❑ Began collecting LIBS data from material samples
- ❑ 20 measurements/sample, 200 averaged spectra/measurement (4,000 laser shots!)
- ❑ Excellent signal-to-noise so far for all elements of interest





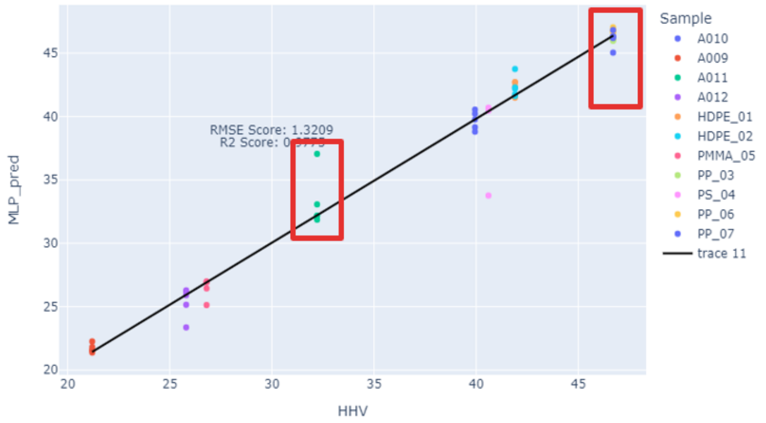


LIBS Signal-to-Noise from Switchgrass (Selected Peaks)

Wavelength	247.86	249.77	259.942	279.522	288.159	323.452	330.294	396.152	422.673	589.00	670.78	777.47	766.49
Element	C I	B I	Fe II	Mg II	Si I	Ti II	Zn I	Al I	Ca II	Na I	Li I	O I	K I
S/N	786.32	7.67	111.51	2030.27	623.51	25.81	8.61	93.04	680.62	262.27	6.94	4.31	831.36

Work on Task 4

- AI modeling underway (decision tree, support vector machine (SVM), random forets, and multi-layer perceptron (MLP) neural networks).
- New approaches being develop to address repeatability of LIBS spectral data (intensity correction approach) and accuracy of prediction.



MLP Modeling Results Compared to Analytical Data

Plastic	HHV
PMMA	26.8
HDPE	41.9
PP	46.7
PS	40.6
PVC	21.2
ABS	39.94
Polycarbonate	32.21
Acrylic	25.8

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