Project Title: “U.S. Coal to Conductive Inks”

DOE/NETL Virtual Project Review Meeting 26-28 April 2021

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Award Grantee: Minus 100, LLC

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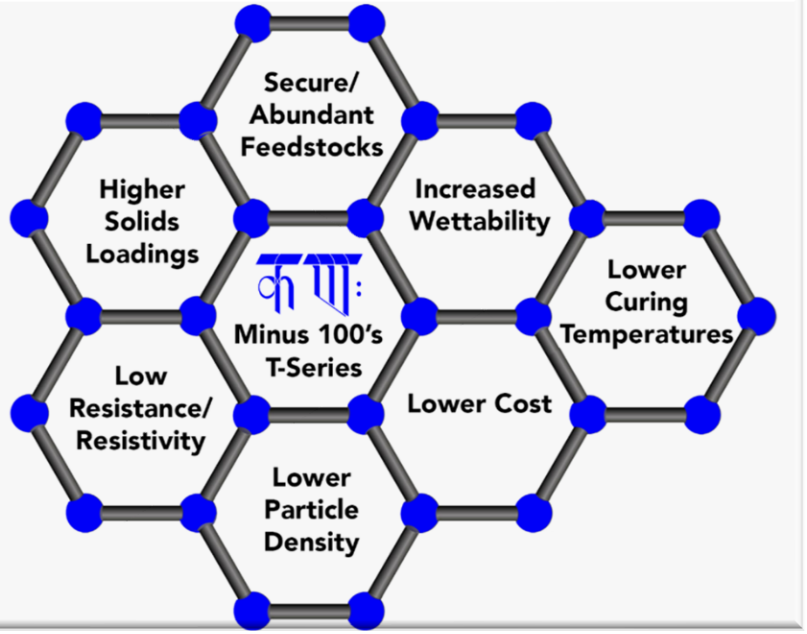
Fossil Energy Objective & Minus 100, LLC Value Proposition

Fossil Energy Objective

- Increase use of U.S. coal utilization through the development of technologies and value-added products that use U.S. Coal Supplies as a primary feedstock

Minus 100, LLC Value Proposition



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Conductive Pigment Markets
(Conductive Inks & Related Markets)

- Conductive Inks
  - Current Market (~ $ 3.1 B/yr) Growing at 3.5% CAGR
  - Conductivity Enhancement Methods Under Evaluation
  - Lower Resistivity Leads to Electronic Printing Market Expansion
- Conductive Paints & Coatings
  - Significantly Larger Market (~ $ 18.7 B/yr) Growing at 6.8% CAGR
  - Application Methods are Simpler
- Synthetic Graphite Manufacturing
  - Significantly Larger Market (~ $ 18.3 B/yr) Growing at 5.2% CAGR
  - Precursor to Various Carbon Allotropes
    - Graphene, Carbon Nanotubes
  - Strategic Material with Limited U.S. Manufacturing Capacity
- Underfloor Heating Market
  - (~$6.3 B/yr) Growing at 4.5% CAGR

Carbon-Based Conductive Ink Applications

(Ref Henkel Web Page)

Sheet Resistivity (ohm/sq/mil)	Printing Method	Potential Application
1,000,000 ± 15%	Screen-printing, thermoset, rigid carbon ink, blending for specific resistance targets	Printed resistors, heaters, potentiometers, friction
100,000 ± 15%	Screen-printing, thermoset, rigid carbon ink, blending for specific resistance targets	Printed resistors, heaters, potentiometers, friction
10,000 ± 15%	Screen-printing, thermoset, rigid carbon ink, blending for specific resistance targets	Printed resistors, heaters, potentiometers, friction
1,000 ± 15%	Screen-printing, thermoset, rigid carbon ink, blending for specific resistance targets	Printed resistors, heaters, potentiometers, friction
100 ± 15%	Screen-printing, thermoset, rigid carbon ink, blending for specific resistance targets	Printed resistors, heaters, potentiometers. friction
< 40	Flexographic, Gravure, Screen, Digital	Printed resistors, heaters, potentiometers, friction
1	Flexographic, Gravure, Screen, Digital	RFID Applications

Position of Minus 100 in the Printing Value Chain

Minus 100, LLC Position in the Conductive Ink Value Chain

Typically Mined Materials,
Domestic Raw Material Supply
Eliminates Reliance on Uncertain Global Conditions

Pigments/Suspensions
Base Vehicles & Additives
(e.g., Binders, Coatings, Preservatives, Dispersants, Curing Agents)

Formulates Inks in Accordance with Printing Method Utilized and Substrate to be Utilized
Paper & Cardboard
Plastic & Metals

Lithographic Printing
Flexographic Printing
Gravure Printing
Screen Printing
Inkjet Ink Printing

Important Electrically Conductive Pigment and Ink Properties

Pigment Properties

- Powder Resistivity (ohm-cm)
- Particle Size (µm)
- Surface Area (m2/g)
- Density (g/cc)

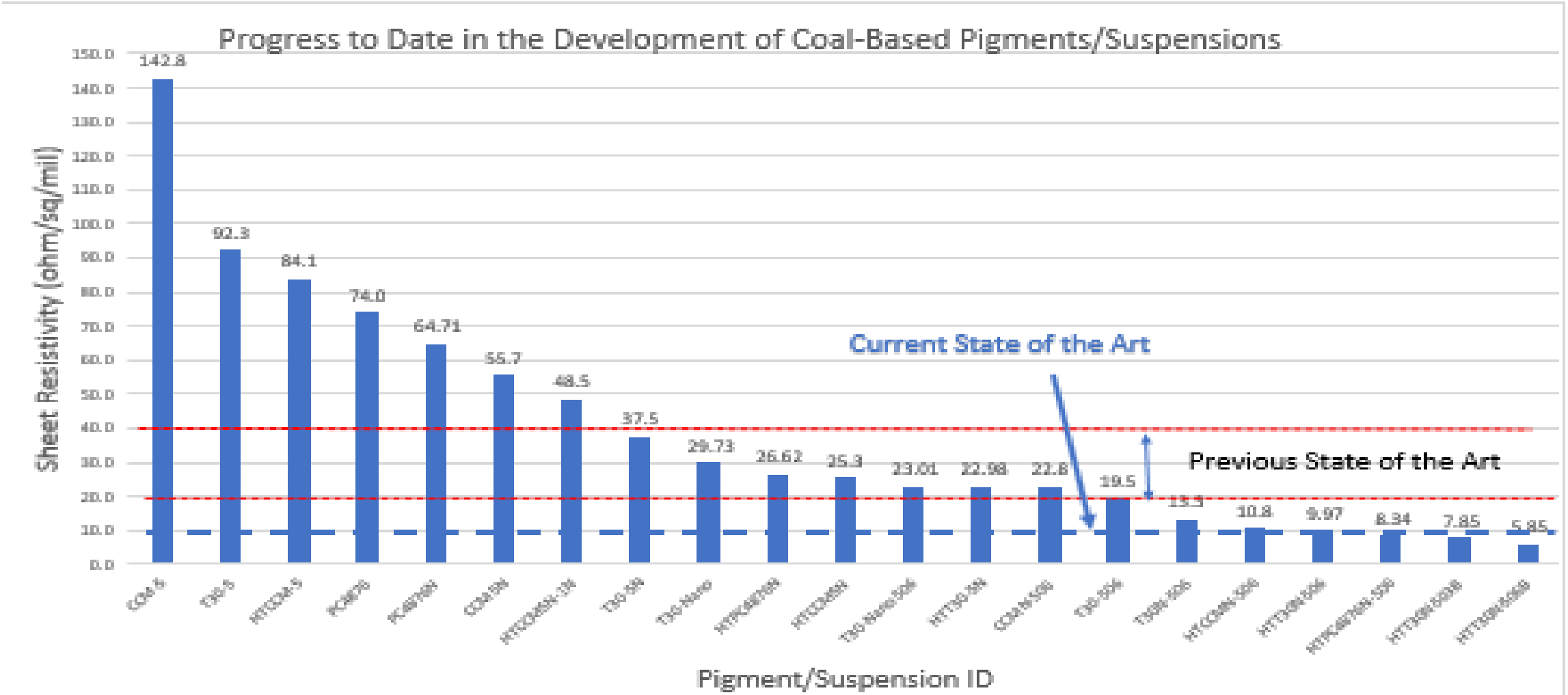
Ink Properties

Sheet/Volume Resistivity (ohm/sq), (ohm/sq/mil)

- Viscosity (Pa·s)
- Surface Tension (dyne/cm)
- Substrate Surface Energy & Surface Adhesion
- Curing/Drying Rate & Temperature
- Abrasion Resistance
- Flexibility
- Color/Transparency
- Toxicity

Technical Accomplishments to Date

(Water Based Ink Formulations)



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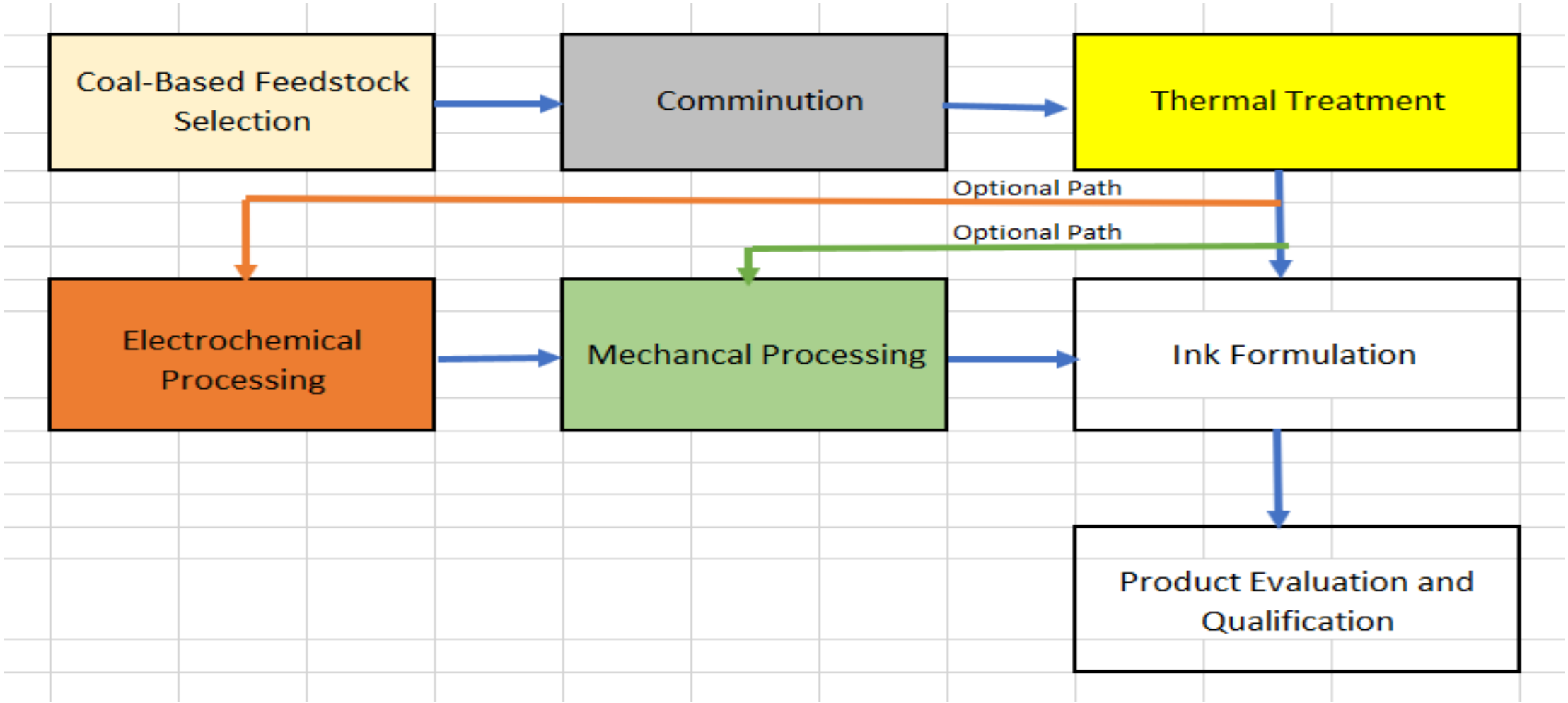
Technology/Product Development Approach



Proprietary Thermal Treatment Processes

Heat Treated Coal-Based Product

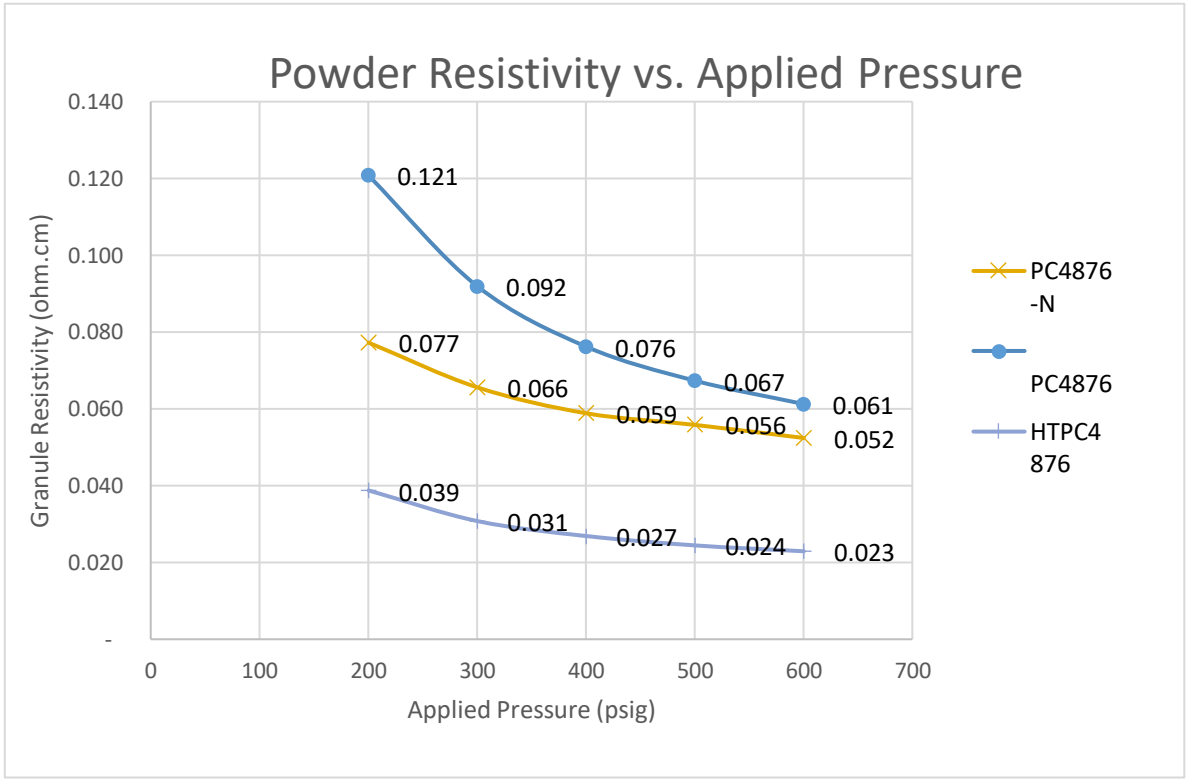
Research Findings

- Elevated temperatures enhance the electrical conductivity of coal-based feedstocks.
- Minus 100, LLC has developed a novel high temperature process for converting coal-based feedstocks into electrically conductive materials.
- This process is suitable for the production of synthetic graphite, a strategic material, from coal-based feedstocks
- Patent application restrictions preclude disclosure of details.

Enhanced Conductivity of Bituminous Coal-Based Feedstocks via Thermal Treatment

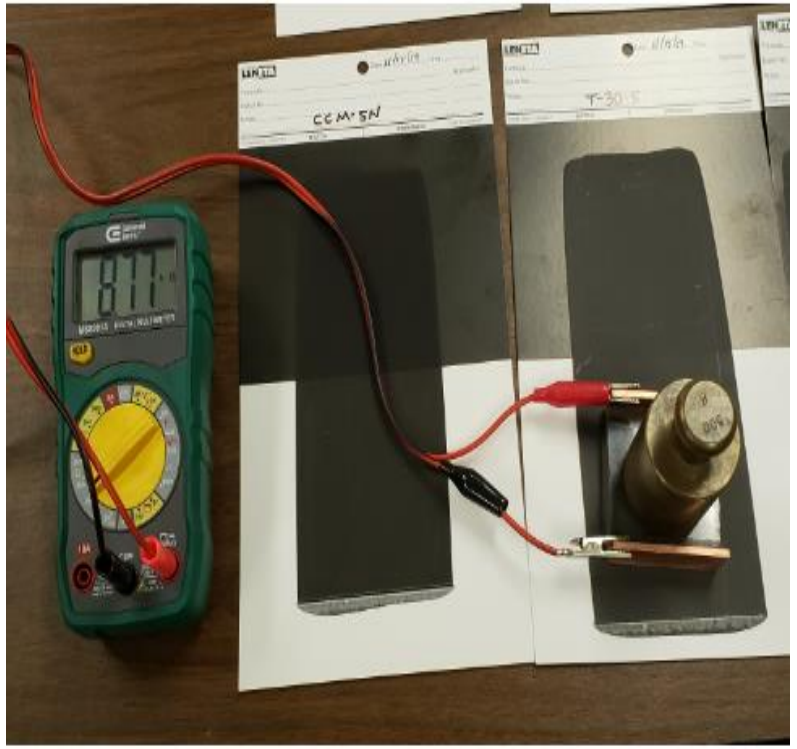
Apparatus for Measuring Powder Resistivity

Powder Resistivity versus Applied Pressure



Ink Sheet Resistivity Measurements

Draw Down with a Mayer Rod



Measurement of Square Resistance

Measurement of Ink Film Thickness

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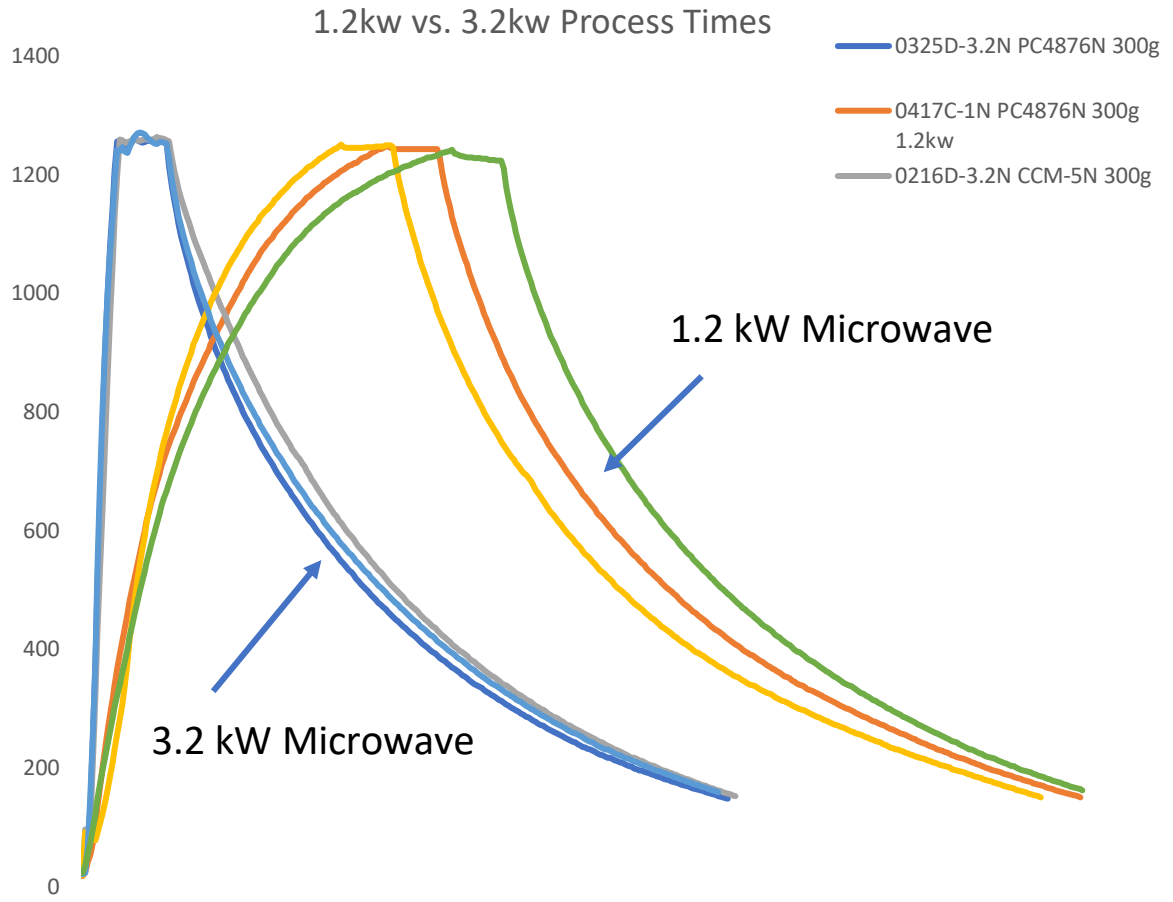
Microwave Processing of Coal-Based Pigments

Performance Comparison of 1.2 kW to 3.2 kW Microwave Furnaces

With the introduction of the 3.2 kW MW, we are observing not only reduced Process Times but Increased Product Yield. In addition, the 3.2 kW MW offers the ability to process at higher temperatures.



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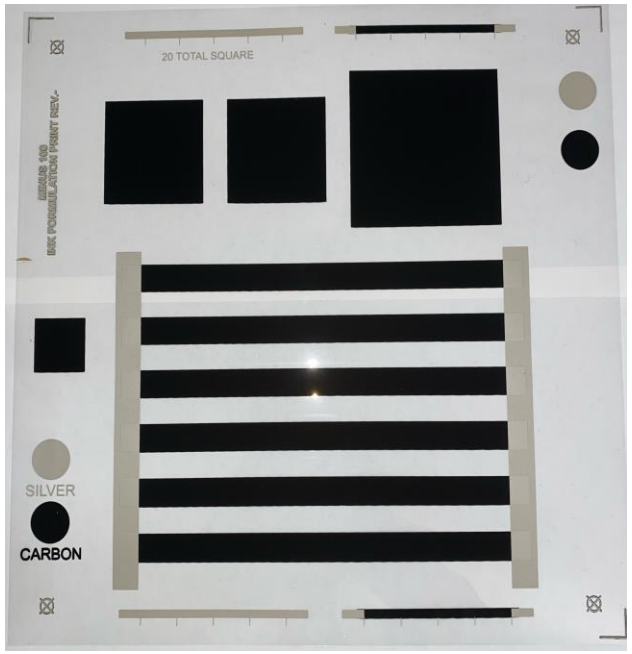
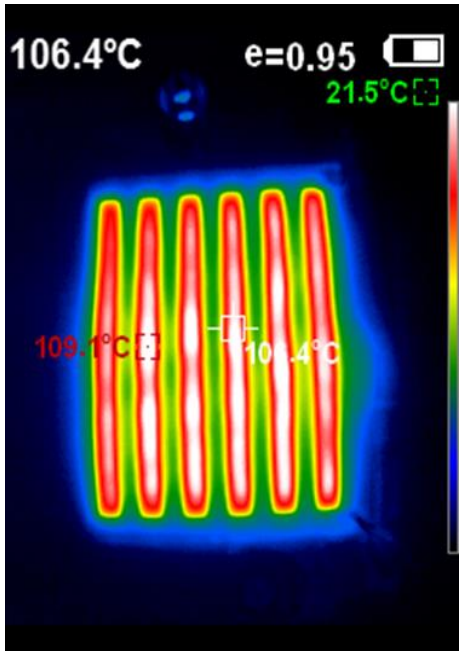
Resistive Heating Element Application

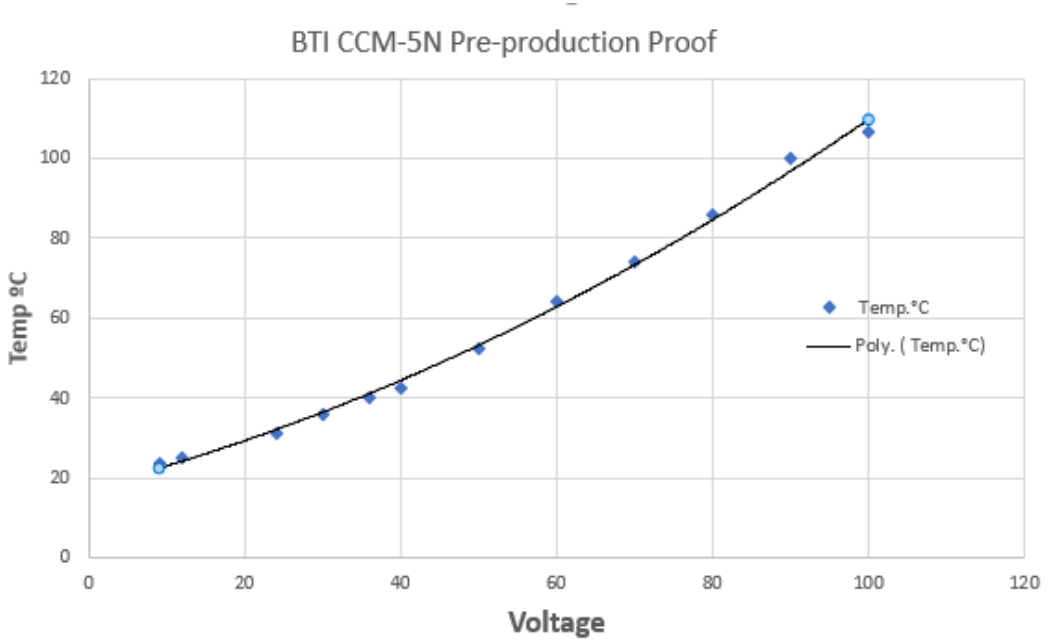
Coal-Based Heating Element Laboratory Pre-Production Proof



Screen Printed Proof Utilizing CCM-5N Pigment From Minus 100 LLC. In a Commercial Grade Screen Ink.
Printed By Butler Technologies



Thermographic Image of Pre-Production Proof
Heating Assembly



Thermoprofile of Pre-Production Proof Heating Element Assembly

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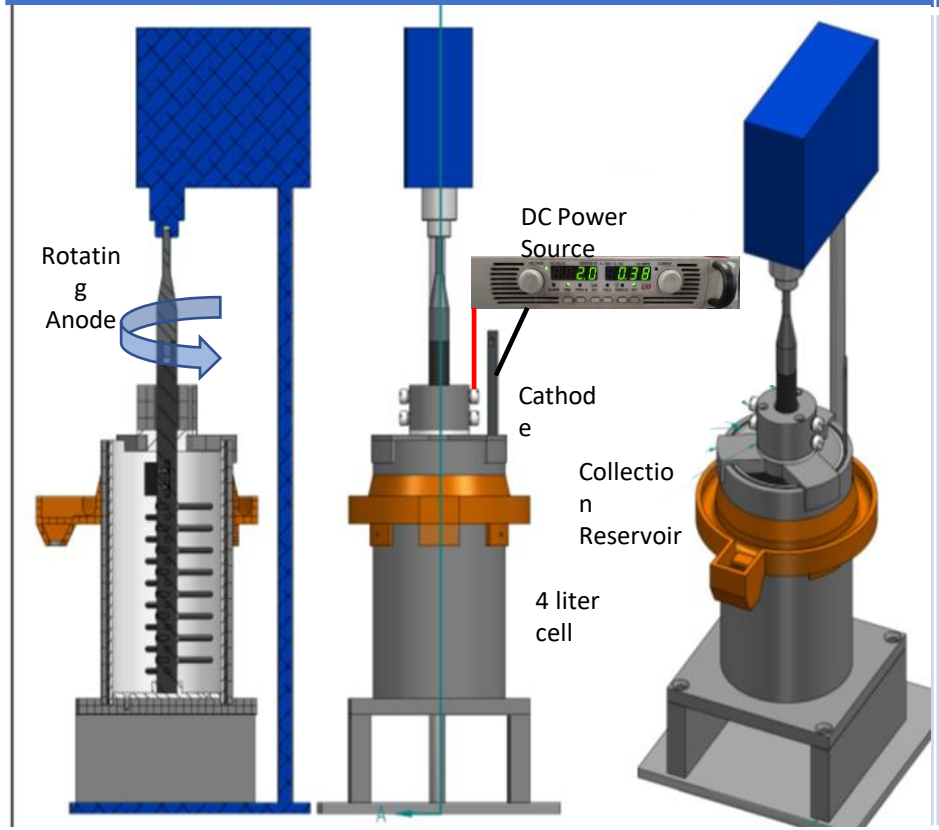
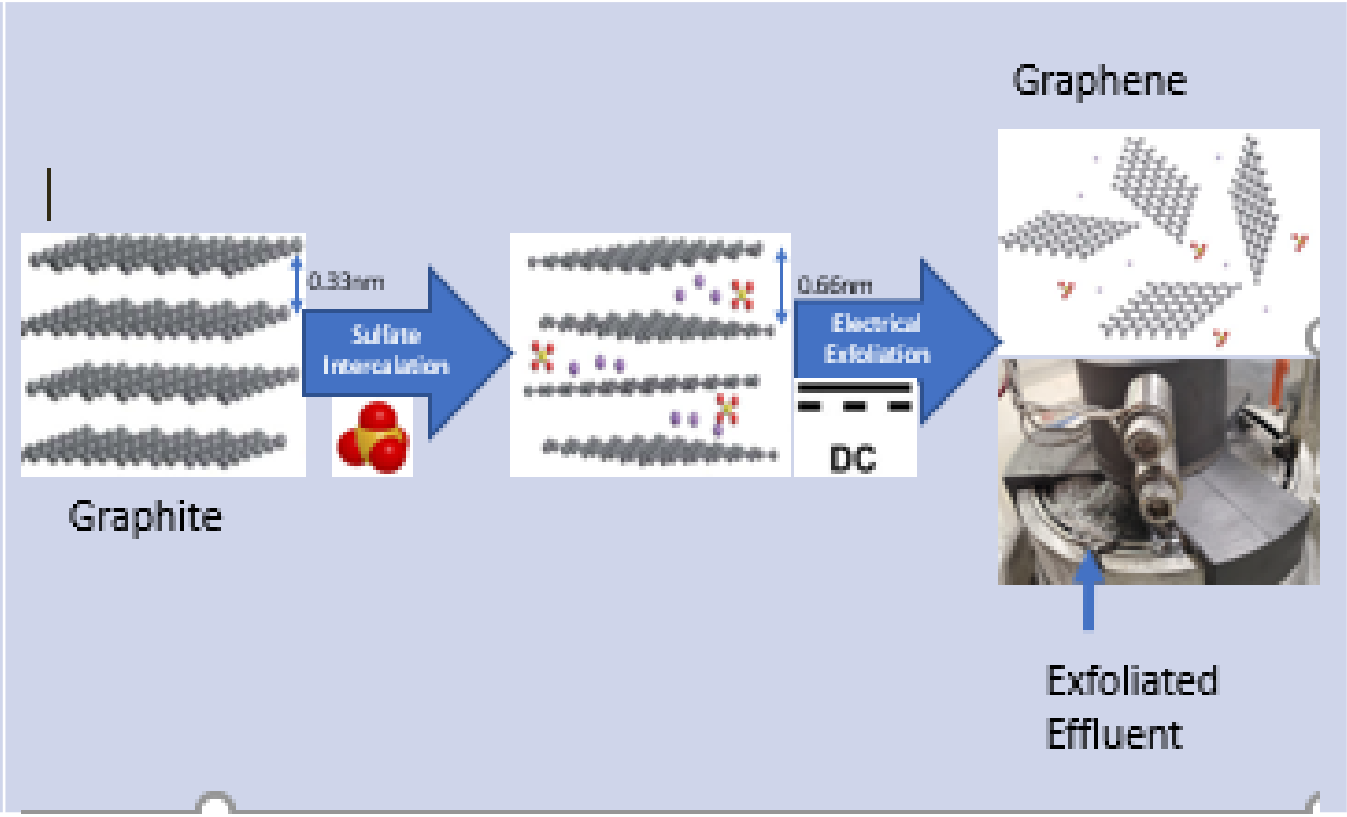
Electrochemical Processing of Coal-Based Pigments

Electrochemical Reactor for Processing Carbon Powders



Intercalation-Exfoliation Mechanism



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Hybrid Metal/Coal-Based Pigment Development

Sample Property	RD24456H	RD24456E ECR	RD24456G	RD24456A ECR	RD24456C	RD24456E ECR
Sample ID	HTCCMN-5	HTCCMN-5E	HTT30N-5	HTT30N-5E	HTPC4876E	HTCCMN-5E
% Carbon	100.00	99.86	99.91	99.90	99.92	99.86
% Moisture	0.02	0.00	0.01	0.01	0.01	0.00
% Sulfur	0.0044	0.0085	0.0036	0.0077	0.0040	0.0085
Electrical Resistivity (Ω*cm)	0.1091	0.1203	0.0672	0.0819	0.0476	0.1203
True Density (g/cc)	2.17	2.16	2.17	2.16	2.21	2.16
Surface Area (m^2/g)	9.68	8.87	15.84	13.55	4.13	8.87
Microtrac (µm)						
MT10 (µm)	1.616	1.6	2.339	1.562	1.901	1.6
MT50 (µm)	5.83	5.92	6.82	5.75	13.23	5.92
MT90 (µm)	12.89	14.55	19.71	15.88	44.64	14.55
MTMV (µm)	6.93	7.37	9.92	8.06	19.44	7.37
MTSTD (µm)	4.13	4.6	5.73	4.95	17.52	4.6
XRD Analysis						
Identified Phase	Graphite(2H)	Graphite(2H)	Graphite(2H & 3R)	Graphite(2H)	Graphite(2H)	Graphite(2H)
002 2-Theta	26.453°	26.443°	26.391°	26.427°	26.409°	26.443°
D-Spacing	3.3667Å	3.3679Å	3.3745Å	3.3699Å	3.3722Å	3.3679Å
Crystallinity	100%	100%	100%	100%	100%	100%
L_a Crystallite size(100→110)	101.74Å	52.43Å	108.13Å	107.56Å	172.30Å	52.43Å
L_c Crystallite Size(002→004)	203.08Å	218.00Å	162.22Å	153.53Å	237.00Å	218.00Å

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Additive Enhancement for Coal-Based Pigments

Experimental Design (DOE) for the Production of Graphite/Graphene Platelets

Factor (X)	Levels	Experimental Trials	Results (Y)
 Graphite Platelets	5wt% 10wt%	All possible level combinations yield **12** trials. Design results will be randomized and analyzed using Minitab Software	Sheet Resistivity Powder Resistivity Particle Size Expansion Fraction
 Na_2SO_4	0.5M 1.0M		
 Intercalation Time @ 2.00V	10min 20min 40min		

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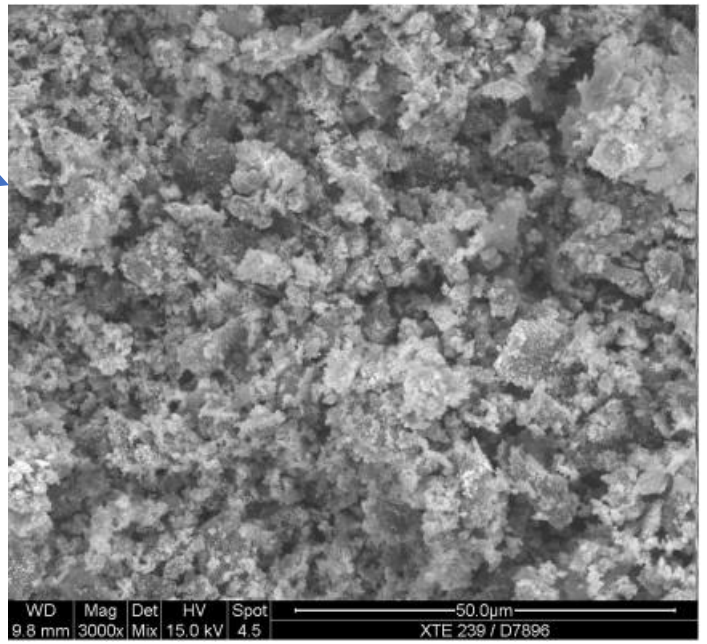
Hybrid Metal/Coal-Based Pigment Development

Metallization of Coal-Based Pigments via Electroless Nickel (EN) Plating

Property / Level of alloy	High[a]	Mid[a]	Mid-Low[a]	Low[a]
% Phosphorous	10 - 13	7 - 9	4 - 6	1 - 3
Electrical resistivity[h]	75 - 110	40 - 70	15 - 45	10 - 30

$$2Ni^{2+} + 8H_2PO^-_2 + 2H_2O \rightarrow 2Ni_0\,(s) + 6H_2PO^-_3 + 2H^+ + 2P\,(s) + 3H_2\,(g)$$

- <u>Stage I</u>
- 5µm coal based conductive particles were successfully coated with nickel alloy.
- Goal to lower ink weight and cost compared to silver.
- Successfully coated agglomerate free
- <u>Stage II</u>
- To date, commercially available chemistry coatings have not achieved published resistivity targets (15-45 µohm-cm).
- Working with two reputable plating companies to increase coating conductivity.
- Investigating phosphorus content of coatings.
- Pursuing surface analysis and oxidation inhibition of coatings.



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SWOT/SCOT Analysis

Strengths

- Phase I Goal: < 1000 ohm/sq/mil - Achieved
- Phase II Goal: < 100 ohm/sq/mil - Achieved
- Internal Goal: < 10 ohm/sq/mil – Achieved 6 ohm/sq/mil
- Next Target: ≤ 1 ohm/sq/mil – In Progress
- Lab-Scale heating element prototype developed
- Development of novel high temperature process for conductive/graphitic enhancement
- Use of nontoxic electrolytes in ECR

Challenges

- Achieve ≤ 1 ohm/sq/mil with metallized pigment.
- HT Furnace construction materials
- Flue Gas Emissions
- Material handling of fine particles

Opportunities

- Seeking underfloor heating commercialization partner
- New electronic circuit printing
- Collaborating with major ink manufacturers
- Conductive Paints and Coatings
- Synthetic Graphite Production
- Semiconductor Chemical Mechanical Planarization (CMP) Spillover

Threats

- Covid-19 Fallout (supply and co-development)
- Demand destruction

Commercialization Activities

- Accomplished Phase II Objective of ≤ 100 ohm/sq/mil with Water and Solvent-based Inks
- Major Ink Manufacturers are Evaluating Coal-Based Pigment & Ink Suspensions for Conductive Ink Applications
- Working Toward Qualification of our Pigments/Suspensions for Specific Commercial Applications
- Testing of Lab-scale Proto-type Heating Element Assemblies
- Working with Commercial Screen Printer to Scale-up Heating Element Assembly Designs
- Developing Lower Resistivity Pigments/Suspensions to Expand the Application Scope of our Coal-Based Pigments/Suspensions

COMMERCIALIZATION EFFORTS (Cont'd)

- Discretionary Commercialization Assistance (DCA) provider: TechOpp Consulting, Inc. (TOC)
- Developing and executing commercialization strategy to enable expansion into commercial markets
  - To date, connected with Major Conductive ink Manufacturers, Paint/Coating Manufacturers and Electric Underfloor Heating Manufacturers/Distributors
- Identifying and pursuing Phase III opportunities within non-DOE federal programs (e.g., DoD)
- Instrumental in securing LOS from Key Industry Players for Phase IIA
- Networking with Top Level Scientists / Business Development Personnel of Large Companies
- Filling the "Gap" for Small Organizations

Future Plans

1. Initiate Phase IIA Project
2. Continue to Pursue electric Underfloor & Wall Heating Element Applications
    - Engaged with co-development commercial screen-printing partner to produce a protype.
3. Continue Electro-Chemical Research
    - Evaluate intercalation/exfoliation potential of selected coal-based pigments.
4. Complete Carbon-based Additive Research
        - Conductive Carbon Black
        - Graphite/Graphene Platelets
        - Carbon Nanotubes
5. Continue Hybrid Pigment Conductive Research
        - Metallization
6. Continue collaboration with major ink formulators to evaluate Minus 100, LLC conductive pigments.
7. Initiate lab-scale testing and evaluation of proprietary heating technology at elevated temperatures
8. Continue commercialization efforts with Tech-Opps.

Acknowledgement & Disclaimer

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Questions & Answers