Form #: T06-13 **Official Test Report**
Initial Release, 11/26/2013

September 4, 2014 Project 14.999

Recycled Fuels, Inc.
123 Main Street
Anywhere, USA 45678

Attn: John Smith
Phone: 555-555-1234
Email: jsmith@rfi.com

Failure Analysis

This report contains the results of characterization of the solid deposits causing blockage inside the RFI reactor and outlet tube. Samples were submitted by John Smith on 8/22/14.

Material Characterization Summary

LCM Sample ID	Client Sample ID	Results
14.999-01a	Shiny, hard region of deposits	**FTIR**: No peaks (suggests elemental carbon) **XRF**: Increased concentration: Ti, Fe **EDS**: C = 93.4% ± 3.6, O = 4.9% ± 3.1, S = 1.2% ± 0.9, Na = 0.2% ± 0.2, Ca = 0.2% ± 0.4, Si and K = < 0.1% **TGA**: 0.0-0.5% hi vol, 1.2-5.5 % med vol, 17.3-19.1% carbon, 76.7-79.7% high temp carbon/ash **Temperature Profile**: No apparent change in material
14.999-01b	Dull, powdery region of deposits	**FTIR**: No peaks (suggests elemental carbon) **XRF**: Increased concentration: Ti, Fe **EDS**: C = 95.4% ± 1.0, O = 3.6% ± 1.1, S = 0.9% ± 0.1, Al = < 0.1% **TGA**: < 0.1-0.2% hi vol, 0.5-0.8 % med vol, 10.8-17.6% carbon, 81.8-88.2% high temp carbon/ash **Temperature Profile**: No apparent change in material

Comments
Samples were analyzed by Thermo Nicolet FTIR, Amptek XRF, Hitachi SEM/EDS, an adapted step TGA method, and a Thermolyne furnace.

FTIR Results
Infrared spectra of both regions of the sample are featureless except for very minor peaks in the 1400-1600 cm^{-1} region (Figure 1) and a peak near 600 cm^{-1} in sample 14.999-01a. The lack of major peaks is indicative of a nearly pure elemental substance, which we can conclude is carbon based on the rest of the results below. The small features are likely due to impurities trapped in the carbon deposits. The relatively weak intensity of the peaks makes it impossible to determine the specific nature of the impurities and suggests they are present only in trace amounts with a slightly higher concentration in sample 14.999-01a (consistent with other results).

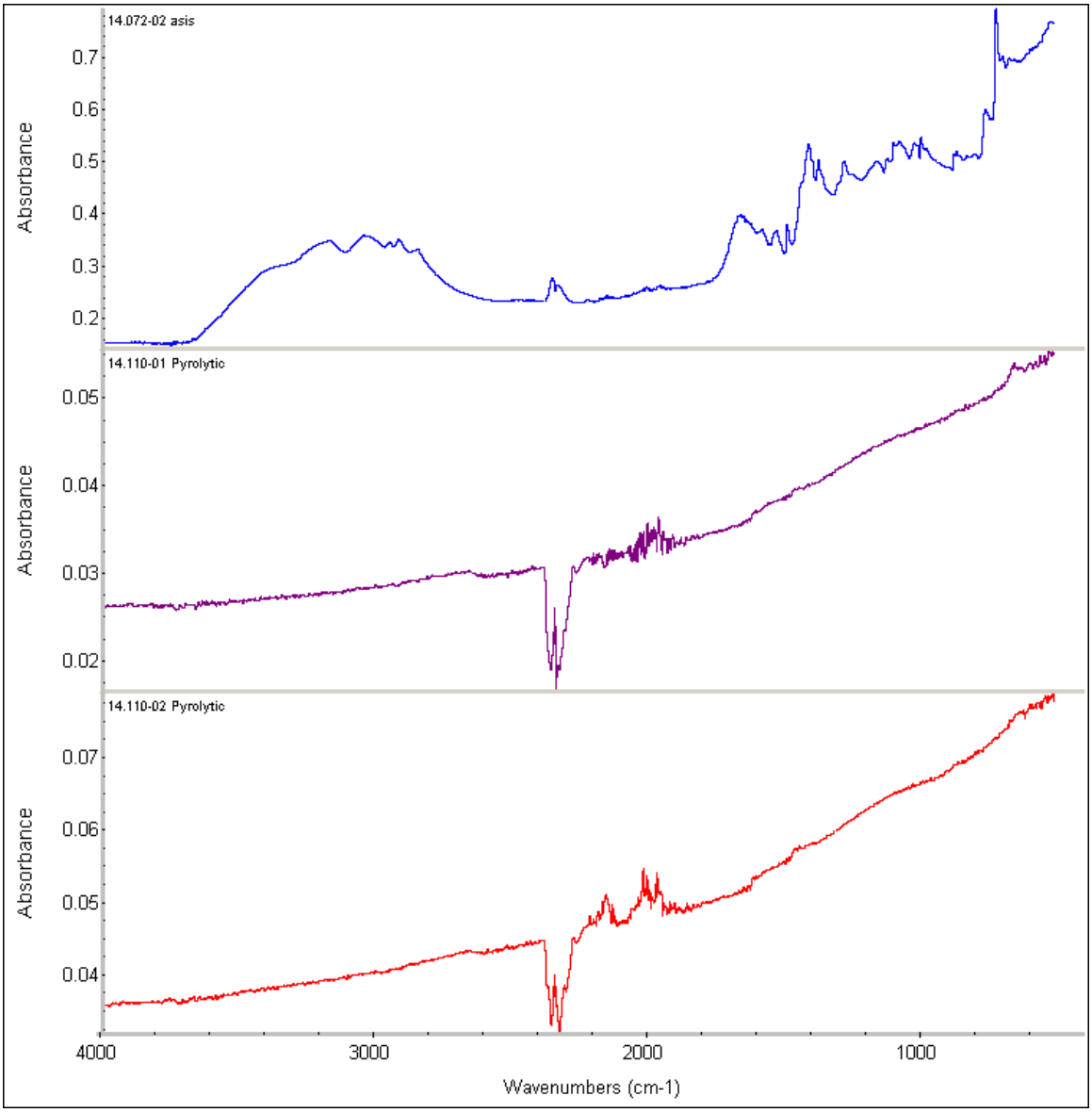

Figure 1. FTIR spectra of asphaltenes (blue), 14.999-01a (purple), and 14.999-01b (red). Note the difference in absorbance scales. The features in the 1900-2200 cm^{-1} region are due to atmospheric interferences from H_2O and CO_2. The figure clearly shows the lack of asphaltene features and the featureless nature of the sample spectra.

XRF Results

Trace amounts of various elements were detected in XRF data and X-ray counts were compared to those from the raw material. Steel elements (titanium and iron) are present at levels that suggest degradation or wear of the reactor structure.

Percent elemental concentration vs. raw material (%)

Sample	Silicon	Sulfur	Chlorine	Calcium	Zinc	Titanium	Iron
14.999-01a	9.0	56.8	0.8	50.9	14.7	403.2	145.1
14.999-01b	2.9	69.7	18.5	75.8	16.1	204.2	118.2

SEM and EDS Results

SEM images of the material show that 14.999-01b has a much smoother structure than 14.999-01a (Figure 2). This difference indicates that the shiny deposits found hotter regions of the reactor have been annealed. The EDS quantitative elemental analysis shows a very high carbon concentration in both samples. Sample 14.999-01b has a higher carbon level, lower oxygen and sulfur levels, and no detectable metals. This serves as further evidence of annealing at high temperature, which would remove oxygen and sulfur.

Atomic percentage of elements detected by EDS (%)

Sample 14.999-01A	C	O	S	Si	Ca
Avg	93.4	4.9	1.3	0.1	0.3
Std Dev	3.6	3.1	0.9	0.2	0.4

Sample 14.999-01B	C	O	S	Si	Ca
Avg	95.4	3.6	0.9	0.0	0.0
Std Dev	1.0	1.1	0.1	0.0	0.0

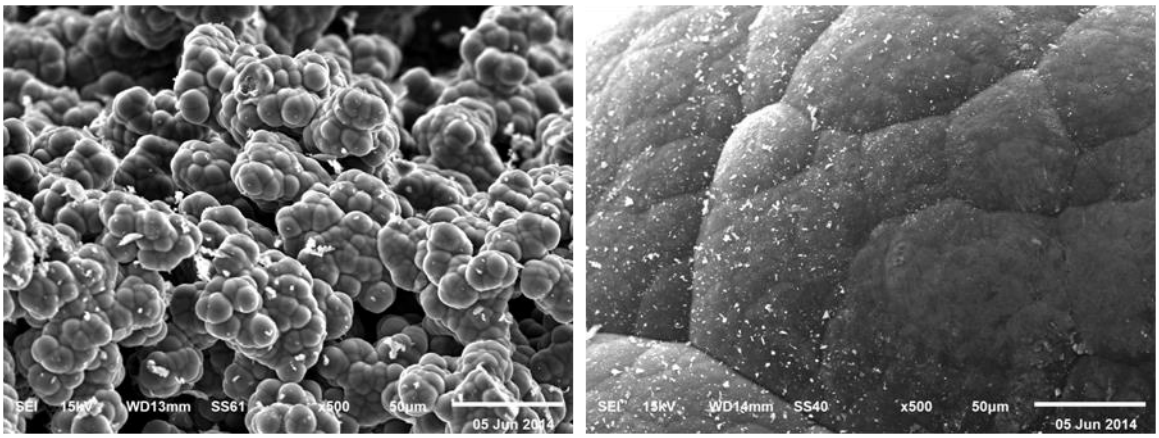

Figure 2. SEM images of 14.999-01a (left) and 14.999-01b (right) show the difference between the rough, granular structure of deposits from cool regions and the smooth structure of deposits from the hotter regions.

Temperature Study Results

The samples were subjected to the following temperatures (°C) under ambient air: 21, 325, 477, 488, 493, 499, 504, 510, 538, 550, 593, 649, and 850. The samples remained solid throughout the experiment, never melting or gasifying to a large extent. The appearance of the samples did not change (Figure 3). The bulk of the material is stable at high temperatures, indicating that the deposits are a highly-ordered, graphite-like structure resulting from the deposition process from gas-phase hydrocarbons and the

subsequent annealing reaction observed in the SEM images. Even the granular superstructure of 14.999-01a is highly ordered on the atomic level.

Percent sample weight lost in each temperature regime (%)

Sample	Highly Volative % (25-325 °C)	Medium Volatile % (325-550 °C)	Combustible % (550-850 °C)	Non-combustible %
14.999-01a 6/3/14	0.5	5.5	17.3	76.7
14.999-01a 6/10/14	0	1.2	19.1	79.7
14.999-01b 6/3/14	0.3	0.8	10.8	88.1
14.999-01b 6/10/14	< 0.1	0.5	17.6	81.8

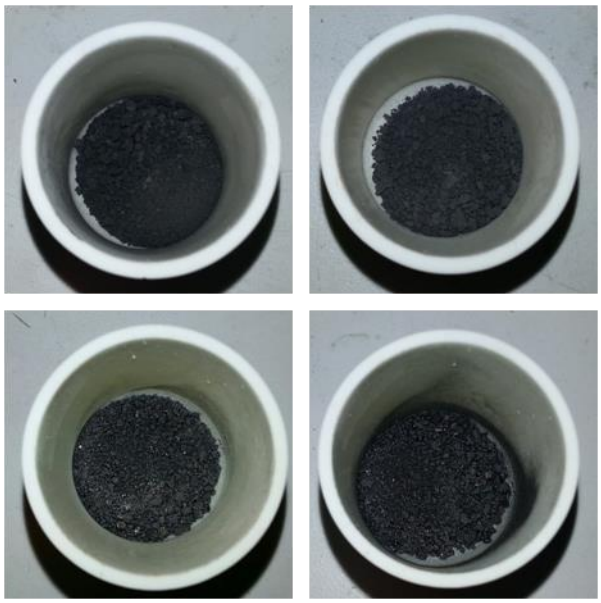

Figure 3. Photos of samples 14.999-01a (top) and 14.999-01b (bottom) show little change in appearance before (left) and after (right) exposure to temperatures as high 850 °C.

Consulting Discussion

The results of the laboratory analyses presented above all indicate that the deposits retrieved from the reactor are high purity elemental carbon with a highly-ordered, graphite-like structure that is resistant to oxidation at high temperatures. These deposits are chemically stable and cannot be removed by the use of higher processing temperatures or the introduction of oxygen into the reactor system. The high purity of the carbon indicates that it is not unreacted raw material also present in the chamber. Rather, the deposits are the result of a chemical reaction between the gases present in the reactor and the stainless steel material of the reactor.

SCIENTIFIC LITERATURE DISCUSSION REMOVED TO COMPLY WITH CLIENT CONFIDENTIALITY AGREEMENT.

Based on previous experience and published reports, we suggest applying a ceramic coating to critical metal surfaces where deposition has been problematic. Two ceramic materials that have been shown to greatly reduce carbon build-up are alumina (Al_2O_3) and silica (SiO_2). This coating should be carefully applied to prevent cracking from heat stress. Furthermore, the ceramic coating will not prevent carbon particles from settling on surfaces. While the metal will remain protected from dusting, these particles can auto-catalyze carbon growth. Therefore, we also recommend the use of mechanical cleaning techniques to remove the carbon particles from the ceramic surfaces. Mechanical techniques that are currently used by RFI may be sufficient.

We recommend X-ray diffraction measurements to determine the crystal structure of the deposits. This will provide valuable information that could lead to a more specific identification of carbon deposits than "pyrolytic carbon".

References and Footnotes

SPECIFIC LITERATURE REFERENCES REMOVED TO COMPLY WITH CLIENT CONFIDENTIALITY AGREEMENT.

The results presented in this report relate only to the samples tested.

This report shall not be duplicated, except in full, without written approval from Lab/Cor Materials, LLC.

Test Conductor	Certifying Manager
Chief Materials Chemist	Chief Scientist

810 NW 45th Street • Seattle, Washington 98107
Phone: (206) 508-1470 • E-mail: mail@labcormaterials.com