PRELIMINARY REPORT

Characteristics of Coal and Char from FMI Process

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Note: The material in this report is preliminary and may be supplemented with additional data at a later time.

Background:

On July 17, Dr. Vijay Sethi, Western Research Institute (WRI), and Dr. Don Dunlop, Fuels Management, Inc. (FMI), demonstrated an oxidative regime coal drying-passivation process at WRI. Cathy Summers, Department of Energy - Albany Research Center, observed the demonstration and collected samples of the feed coal, char products of the process, and fuel oil used in the second stage of the test. The demonstration was made at the request of Tom Sarkus and Leo Makovsky, DOE. FMI has proposed to build a demonstration plant of the process, and is seeking funding from DOE. DOE is looking for evidence that the char produced is not only dry, but is truly passivated; i.e., it has a lowered predisposition to self-heat in the presence of oxygen, and to spontaneously combust as a result.

The process:

A bed of coal is fluidized using a mixture of heated air and recycled off-gas in the reaction vessel. Air is drawn in from the atmosphere, heated with a propane flame, then mixed with the somewhat cooler recycle gas, which has passed through a cyclone to remove solids. Coal is fed from a hopper into the bottom of the bed via a screw feeder at rates between 0 and 400 lb per hour. The residence time of the coal averages between 5 and 7 minutes, and the char is discharged after treatment into sealed 55-gallon drums which have a flow of N_2 going into them.

During the demonstration on July 17, during the first hour after conditions had stabilized at a treatment temperature of 600°F, coal alone was fed into the reactor. A sample of this product was collected (referred to in this report as “Char product 1”). Subsequently, a small amount of fuel oil was injected for a period of time with the coal; the product of this portion of the test was also sampled (“Char product 2"). Dr. Vijay Sethi reported that the fuel oil line was constricted during this part of the test and the amount added was approximately 1.5 cc/3 lb. coal (40 to 50 times less than desired).

Sampling:

Ideal sampling conditions would have consisted of sample discharged directly into an inert-atmosphere environment with no opportunity for oxygen contamination, with all preparations for shipping completed in this environment.

Products of the test were discharged into sealed 55-gallon steel drums with nitrogen gas flowing into them. Once sufficient sample had been discharged into a drum, it was disconnected from the system and its lid (which was modified to attach via a closed spout directly to the top of the reactor) was switched with another that had been modified to include a gas inlet port. A gas line from an argon supply was attached to this port, and the sample cooled under an argon flow. The rate of flow is not known, and because there was only one Ar source, after the second sample was taken, the first was disconnected from the gas line and allowed to continue cooling in whatever atmosphere had been created by the consecutive additions of process off-gas (about 7.5% oxygen and carbon oxides), nitrogen, and argon.

Once cooled, the gas line was disconnected and the lid was removed carefully, attempting to avoid mixing of the external atmosphere with the heavier argon in the drum. Plastic zip-closing bags and a scoop were lowered into the drum and samples weighing approximately 500 g. were placed in the bags. The bags were closed while still in the drums, then removed and bagged in a second plastic bag with an argon gas line feeding into it to generate positive pressure. Excess gas was expressed while sealing the bag. The double-bagged sample was placed in a one-gallon paint can and the argon line was fed into the can while the folds of the bag were manipulated to try to expose and flush pockets of air. The lid was placed on the can, and a wooden mallet was used to close it. Finally, the rim of the lid was sealed with a "quick-drying" caulk.

The feed coal was collected in a similar manner. It was stored at WRI in large drums lined with plastic, having been crushed and screened to 1/4 by 1/8 inch in January, 2000. Since this material had been stored in air, and was open to the air while the demonstration was going on, an attempt was made to flush the sample by feeding argon into the inner bag for a few minutes before sealing it. Again, it was bagged in a second bag with an argon purge, then into a paint can.

Two samples each of feed coal, Char Product 1 (generated **without** added fuel oil), and Char Product 2 (generated **with** fuel oil) were collected. Two samples of the fuel oil, approximately 200 ml each, were also collected.

Analysis:

The analyses shown in Figure 1 were planned.

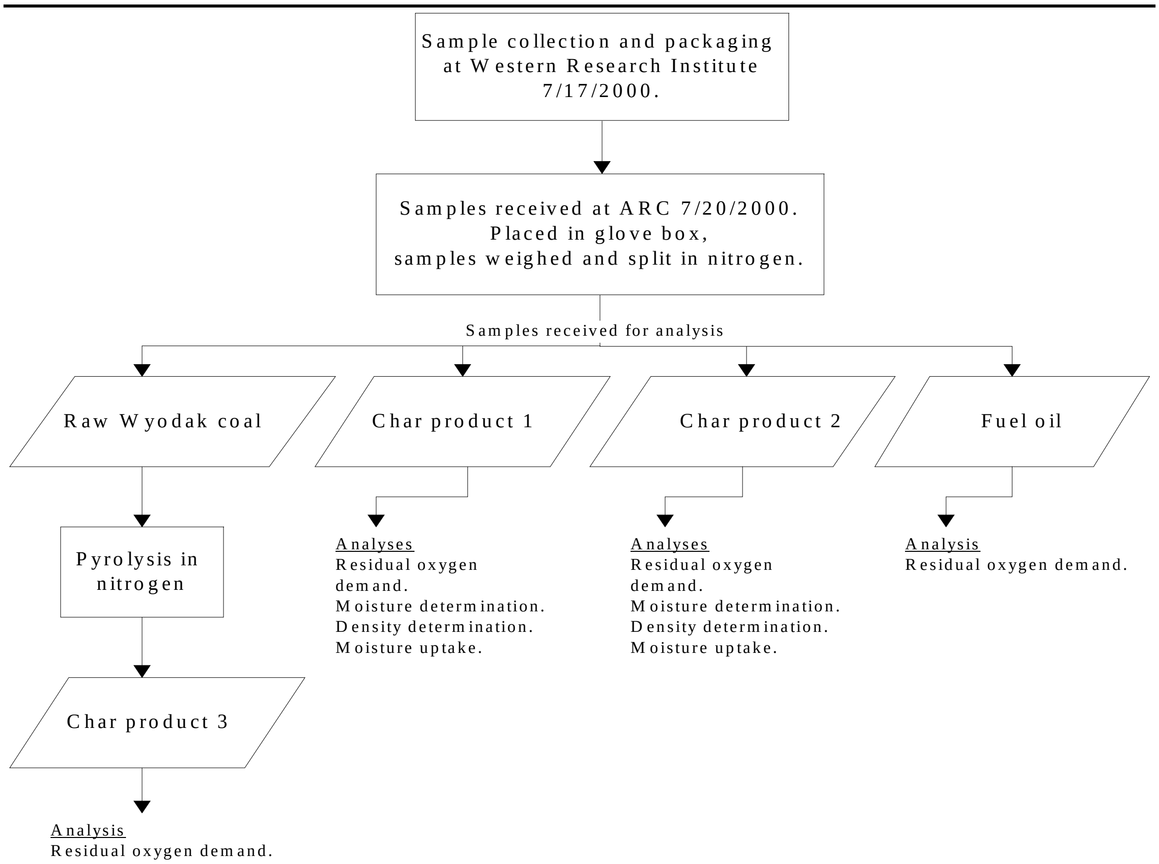

Figure 1. Planned analyses for coal and char products.

To date, at least one residual oxygen demand (ROD) determination, based on a technique developed by the U.S. Bureau of Mines in 1990[1], has been completed for each of the solid samples - feed coal, Char Product 1, and Char Product 2. In addition, the feed coal was pyrolyzed at ARC in nitrogen at approximately 750°F to create a separate product for analysis. During pyrolysis, this coal lost 46% of its weight. The ROD of this material, Char Product 3, has been determined as well.

Moisture determinations are being made by the analytical laboratory at ARC.

Density determinations have been made for the feed coal, and for Char Products 1 and 2. These three samples are also being held in an atmosphere of 80% humidity to determine their abilities to reabsorb moisture.

Results:

Figures 2, 3, and 4 show the results of ROD determinations on the feed coal and the three char products. The materials had final adsorbed oxygen measurements as shown in Table 1.

Table 1. Results of ROD analyses

Sample Identification (ARC number)	Source of Material	Number of Analyses	Average ROD, torr/g
ME3779A	Feed coal	2	3.4
ME3780A and B	Char Product 1	3	8.8
ME3781A and B	Char Product 2	4	9.6
ME3779A-pyr	Char Product 3	1	30.6

Figure 5 shows the results of helium density measurements for the feed coal and the two char products generated at WRI. The three measurements done for the feed coal were consistent, and average 1.33 g/cc. The measurements for the char products, on the other hand, trended strongly downward, probably due to outgassing of volatiles. The fuel oil treated char (Char Product 2) had a greater change through the 4 measurements than did Char Product 1. The density trend lines converge near 1.60 g/cc, and this is probably close to the true densities of these two samples.

Preliminary moisture uptake results are shown in Figure 6. The slopes shown by the two char sample curves are similar, and apparently will approach zero at about a 10% moisture increase, equivalent to an approximate final moisture content of 9.5%.

Summary and Discussion:

Sampling conditions during the demonstration were not ideal for minimizing the exposure of the materials to oxygen. Two packages of each type of sample were shipped to ARC for testing. ROD measurements were consistent between the two packages, indicating that they had similar histories with respect to oxygen exposure. Sampling was probably not a major factor in the final oxygen demand characteristics of the samples.

The ROD results (Table 1, Figures 2, 3, 4) indicate that the feed coal is the least active (has the lowest tendency to self-heat) of the four solid materials tested. This is as expected; wet raw coals are less likely in general to spontaneously combust than coals dried by pyrolysis. The two products produced at WRI are somewhat higher in activity, but less active than the pyrolyzed char produced at ARC as a comparison. The process demonstrated at WRI passivates the coal to some degree. ROD values of 5-10 torr/g have been associated in the past with other coals and chars that have low activities; values of 30 and above are considered very active.

Density measurements are consistent with drying of the char relative to the feed coal.

Moisture uptake measurements show that the chars will reach an equilibrium moisture content near 10%; measurement over a longer period of time will generate more accurate numbers.

Conclusions:

In comparison to other processes, and based on the data in this report, the FMI process has the potential to generate upgraded char material that has a lowered tendency to self-heat and spontaneously combust. In order to best judge the process in comparison to others, it would be necessary to test a greater number of pyrolysis techniques.

No significant difference can be seen as a result of the fuel oil addition. The amount of fuel oil added during the demonstration was much less than intended, and the char therefore is not representative of the process. Dr. Sethi has offered to run another test and generate a char with more fuel oil, if needed.

Although analyses of water content have not been received from the ARC analytical laboratory, typical Powder River Basin coals contain 25-30% moisture. The density differences between the char and the coal indicate a loss of moisture during char production. Assuming that the chars are nearly dry, preliminary results of the reabsorption test show that they will reach a moisture content of about 10% over time.

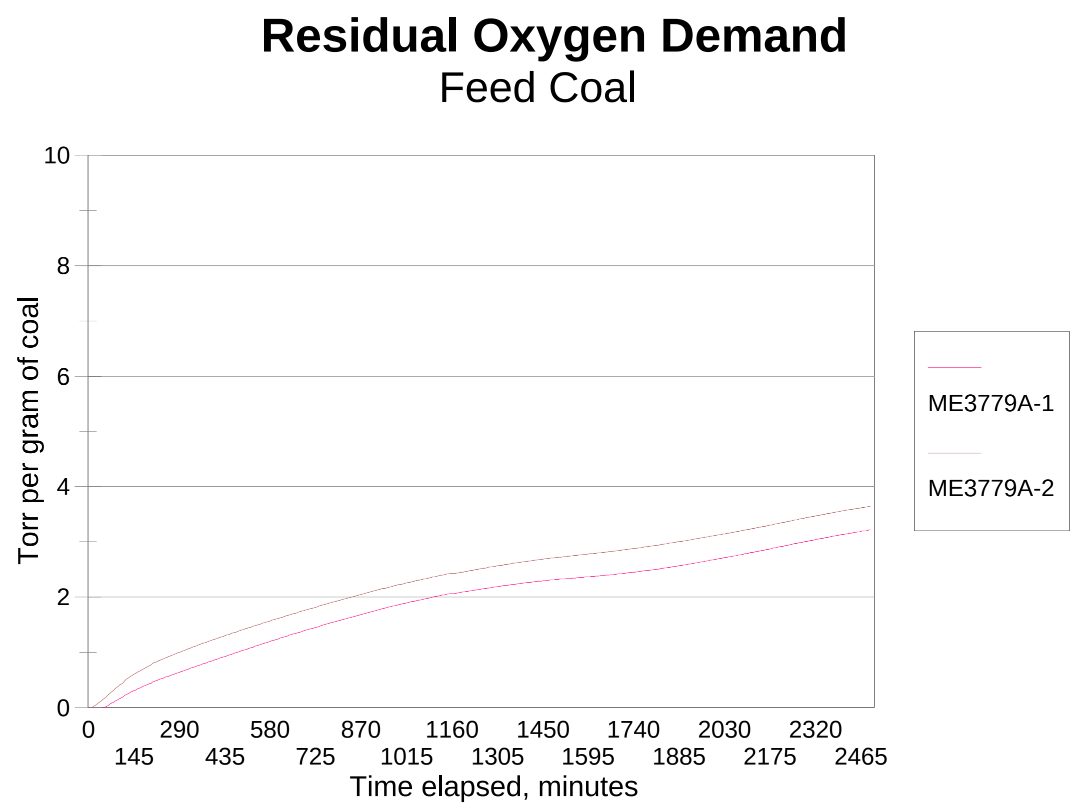

Figure 2. Oxygen absorption of feed coal over time. Values at 2500 minutes are less than 4 torr/gram.

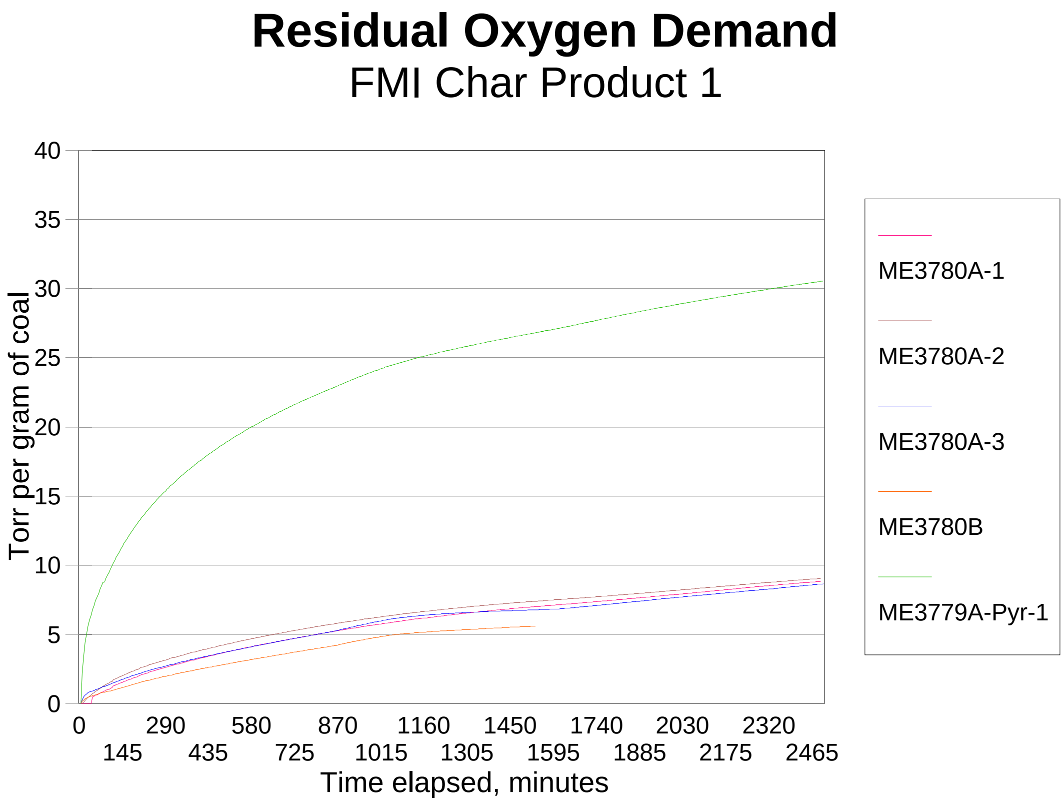

Figure 3. Oxygen absorption of Char Product 1 over time, as compared to Char Product 3 (top line), which was produced by pyrolysis at ARC.

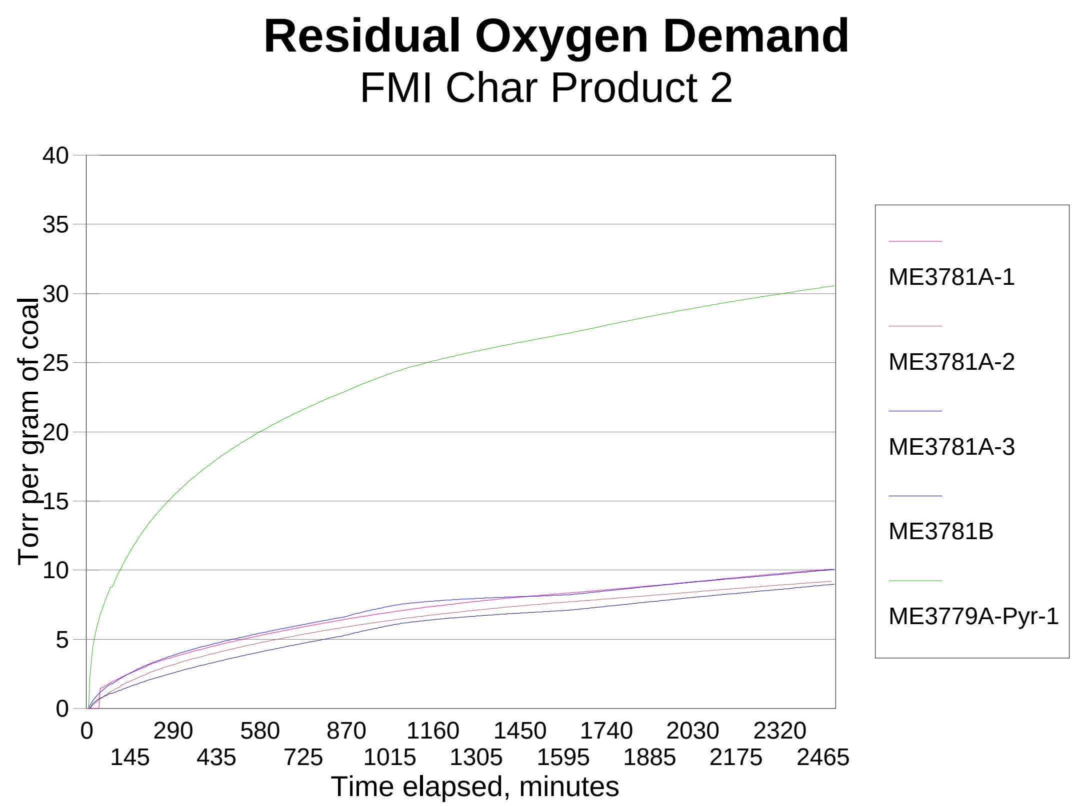

Figure 4. Oxygen absorption of Char Product 2 over time, as compared to Char Product 3 (top line).

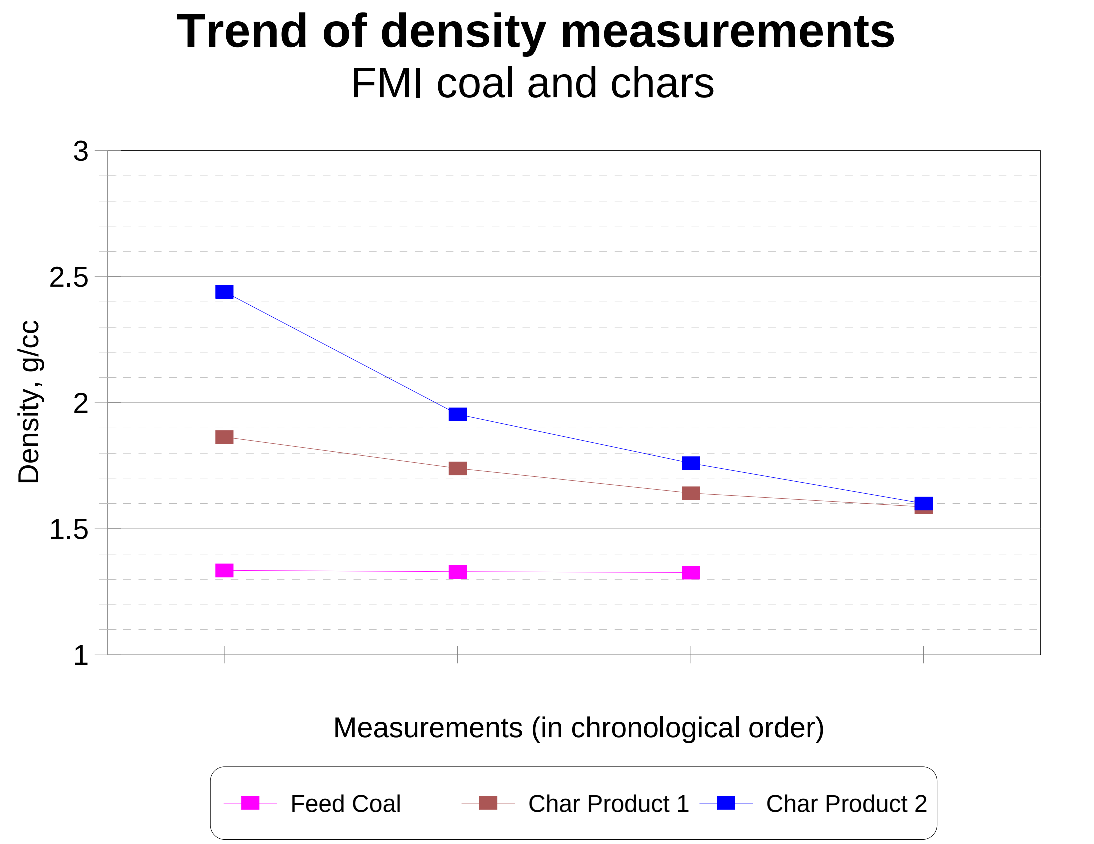

Figure 5. Helium density measurements. The downward trends for the two chars are due to outgassing of volatiles.

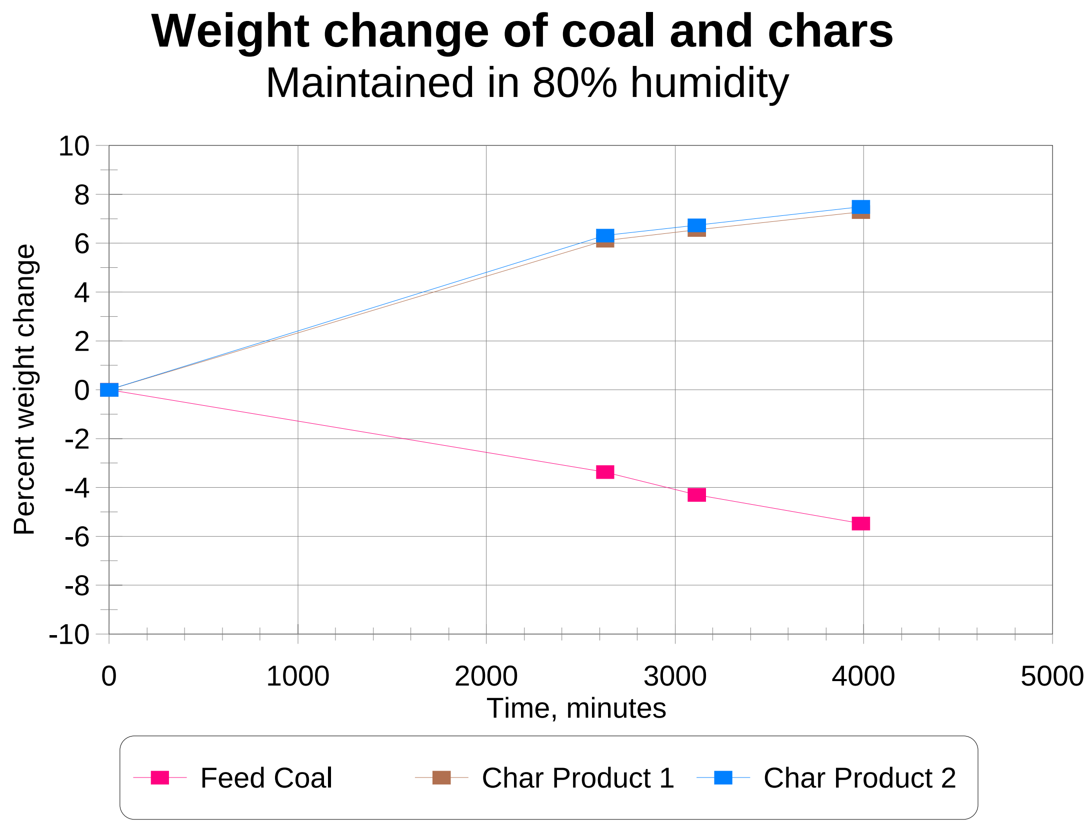

Figure 6. Weight change in coal and chars over time.

1.
Miron, Yael, Alex C. Smith, and Charles P. Lazzara, *Sealed Flask Test for Evaluating the Self-Heating Tendencies of Coals*, U.S.B.M. Report of Investigations 9330, 1990, 18 pp.