

THE USE OF THE BRABENDER PLASTOGRAPH IN STUDYING
THE RHEOLOGY OF ELECTRODE MIXES

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Introduction

The performance of the continuous, self-baking Soderberg electrode, which is widely used in the production of primary aluminum, is closely related to the rheology of the carbon mix that is added to the top of the electrode. The fluidity must be sufficient to ensure an even distribution of the mix over the top of the electrode and to permit the mix to fill voids that are created when contact studs are removed. However, separation of the binder from the aggregate or difficulty in containing the mix in the sheet metal casing may result if the mix is excessively fluid.

Some investigators have attempted to obtain mixes with proper fluidity by studying the rheology of the binders with the assumption that the rheology of the mix is a function of the rheology of the binder.¹⁾* Others have investigated the rheology of the mix itself.²⁾ The latter approach appears to be the more desirable because interactions between the binder and aggregate influence the rheological behavior of the mix.

A method in which a Brabender Plastograph is used for measuring the consistency of the Soderberg mix was described at a previous meeting of this division.³⁾ In the development of the method at the Applied Research Laboratory of the U. S. Steel Corporation, a petroleum-coke aggregate was used that had a smaller size-consist than is commonly used in plant practice. The method has been extended to include the measurement of the consistencies (rheological measurements) of Soderberg mixes containing plant-scale aggregates.

The present paper presents typical results that were obtained in an investigation to (1) use the consistencies of mixes prepared with plant-scale aggregates to predict the optimum binder content for optimum electrode properties and (2) determine the influence of temperature and time in the Soderberg electrode on the consistency of the mix.

Experimental

The two electrode binders and the plant-scale petroleum-coke aggregate used in this investigation are representative of the materials in use at a carbon-paste plant. Some of the more common properties of the two binders are shown in

* See References.

Table I. The properties of the two binders are similar; however, Pitch B contains slightly more benzene- and quinoline-insoluble matter than Pitch A. Past-plant experience has shown that subtle but significant differences exist in the performance of the two binders: pastes prepared with Pitch B required the use of more binder for optimum electrode performance than Pitch A, and the electrodes produced with Pitch B performed better than the electrodes from Pitch A.

The plant-scale petroleum-coke aggregate was graded into the seven fractions shown in Table II. The appropriate weight of each fraction, corresponding to the percentage shown, was used in each binder-aggregate mix.

The major phases of the present investigation are as follows:

1. Determination of the maximum mix consistency for optimum binder concentration and optimum electrode properties. (The two binders were used in the preparation of mixes containing from 28 to 35 percent binder).
2. Investigation of the effect of mixing temperature and holding time on the mix consistency and electrode properties.

The Brabender Plastograph, shown in Figure 1, was used to prepare Soderberg mixes and measure their consistencies. A brief description of the operation of the instrument is given below. The sigma blades in the mixing head are driven by a dynamometer, which is suspended between floating bearings. The torque produced as the blades rotate in the mix at a constant rate of shear is transmitted to the dynamometer. The dynamometer translates the torque through a series of balance levers to a direct-reading balance, which is calibrated to indicate the torque in meter-gram units. A strip chart provides a continuous record of the consistency in terms of meter-gram units. Excessive movement of the lever system is dampened by an oil dash pot.

The mixing head has a working capacity of 650 milliliters. It is heated by recirculating hot oil from a constant-temperature bath through a jacket that surrounds the mixing head. A special insulated lid, not supplied by the instrument manufacturer, minimizes the loss of heat from the head and is an indispensable aid in maintaining the mix at a uniform temperature. Through a small opening in the lid, coke additions can be made without removing the lid.

In the determination of maximum mix consistency for optimum binder concentration and optimum electrode properties, mixes containing from 28 to 34 percent of Pitch A and mixes containing from 30 to 35 percent of Pitch B were prepared at 155 C. In all tests, the weight of the mix was held constant at 700 grams.

In the preparation of a typical mix, the calculated amount of binder is added to the preheated mixing head and is allowed to melt for eight minutes. The binder is then mixed for seven minutes to permit temperature equilibration. The 0.525-inch to 3-mesh fraction (preheated to 155 C) is then added through the opening in the insulated lid. The remaining fractions are added in the order of decreasing size at 5-minute intervals. This sequence of coke additions is used to (1) permit thorough wetting of the large coke particles before the addition

of fine particles to prevent uncoated fine particles from plugging the pores of the large particles and (2) minimize preferential absorption of binder by the fine particles. Mixing is continued for 30 minutes after the addition of the last coke fraction. The torque reading, in meter-grams, at the end of this mixing period is recorded as the consistency of the mix.

The temperature in the Soderberg electrode ranges from about 950 C at the lower working face to about 150 C at the top. As the electrode is consumed, it is lowered, and the unbaked mix in the upper end of the electrode is subjected to gradually increasing temperatures. To provide an indication of the effect of increasing temperature on the consistency of the mix and on the electrode properties, four mixes were prepared at temperatures between 155 C and 225 C. The optimum concentration of Pitch A was used with the mixing procedure previously described.

In the Soderberg electrode, temperature changes occur gradually, and a given portion of the unbaked mix may be subjected to a specific temperature for a relatively long period of time. The effect of time without mixing on the consistency of the mix was determined by repeating consistency measurements on the two mixes with the optimum concentration of Pitch A at 155 C and 225 C. At the conclusion of the normal mixing time, the mixer was stopped, and the mix was maintained at the mix temperature until the consistency approached the limit-of-scale value of 1000 meter-grams or 24 hours, whichever was shorter. Consistency measurements were recorded hourly.

All mixes were packed into perforated graphite molds and baked to 1000 C at a controlled rate of temperature rise in 24 hours. The baked electrodes were then tested for crushing strength and electrical resistivity. The procedure for baking and testing specimen electrodes has been described by Jones, Simon, and Wilt.⁴⁾

Results and Discussion

The relationship between the mix consistency and electrode crushing strength at various binder concentrations is shown in Figure 2. To illustrate this relationship, the experimental data are plotted in bar-chart form. The lined bars represent the consistency of the mix at various levels of binder concentration, and the dotted bars show the crushing strength of specimen electrodes from those mixes. The number at the bottom of each bar represents the percentage of binder in the mix.

This chart indicates that the Plastograph is sufficiently sensitive to detect changes in binder concentration as small as 1 percent and that a good correlation exists between the mix consistency and the electrode crushing strength. As the percentage of binder increases within the limits shown, the consistency and crushing strength values pass through a maximum simultaneously. This relationship indicates that the mix-consistency measurement can be used to predict the optimum binder content for optimum electrode crushing strength. For Pitch A, the optimum binder content for optimum electrode crushing strength is 32 percent and for Pitch B, the optimum binder content is 34 percent. These results correlate well with carbon-paste-plant data on these two binders.

The relationship between the mix consistency and the electrical resistivity of specimen electrodes is shown in Figure 3. A good correlation also exists between these parameters. For each binder, as the consistency values pass through a maximum, the resistivity values pass through a minimum. This relationship further substantiates the premise that the mix-consistency measurement can be used to determine the optimum binder content for optimum electrode properties.

The effect of temperature on the mix consistency and electrode crushing strength is illustrated in Figure 4. The mixes, prepared with 32 percent of pitch A, show successive decreases in consistency as the temperature of the mix increases. The decreasing consistency results from increased fluidity of the binder at the higher temperatures. Simultaneously, the electrode crushing strength increases with higher mix temperatures and lower consistencies. The increase in strength is particularly pronounced between 200 C and 225 C. The higher crushing strengths apparently result from the increased fluidity of the mix. The method described here would provide a suitable means of determining the temperature susceptibility of mixes prepared with various binders.

A susceptibility index (SI) could be calculated with the following equation:

$$SI = \frac{(\text{Consistency at } 155 \text{ C}) - (\text{Consistency at } 225 \text{ C})}{\text{Consistency at } 175 \text{ C}}$$

A low value for the susceptibility index (approaching zero) is indicative of a mix that is not sensitive to temperature change, whereas a high susceptibility index would be obtained with a temperature-sensitive mix.

The effect of hold time without mixing at two temperatures on the mix consistency and electrode properties is shown in Table III. When the mix is maintained at 155 C for 7.5 hours, the mix consistency increases from 600 meter-grams to 1000 meter-grams. Substantial improvements in the electrode crushing strength and electrical resistivity are noted. Similarly, at 225 C, the mix consistency increases from 190 meter-grams to 600 meter-grams in 24 hours, with significant improvements in electrode properties. The increased consistency and improved electrode properties resulting from extended time at high temperatures may be due to an aging or curing of the binder that is initiated or accelerated by the presence of the carbon aggregate. Similar effects probably occur in the Soderberg electrode as the unbaked mix is subjected to elevated temperatures for extended periods of time.

The rheology of the mix in the upper portion of the Soderberg electrode is strongly influenced by temperature and by the length of time the mix is exposed to elevated temperatures. Increased temperature tends to decrease the mix consistency, whereas an extended holding time without mixing at elevated temperatures tends to increase the consistency. The over-all effect of both factors is to improve the electrode crushing strength and electrical resistivity.

Summary

Consistency measurements on Soderberg mixes containing a plant-scale aggregate have been made, with a Brabender Plastograph. The consistencies were

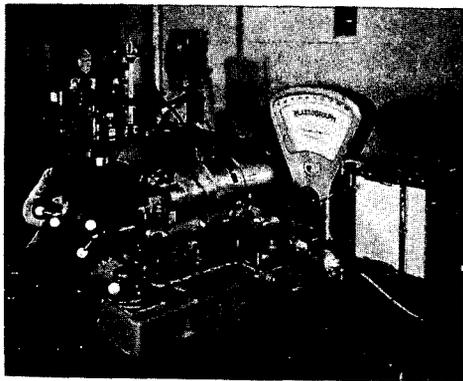
used to predict the optimum binder concentration for each of two binders. A different amount of each binder was required for optimum binder content and optimum electrode properties. This difference was verified by carbon-paste-plant data on the two binders. The effects of temperature and time on the mix consistency and electrode properties were also studied. Increased temperature tends to decrease the mix consistency, whereas consistency increases with an increase in holding time without mixing at elevated temperatures. The over-all effect of the two factors is to improve the electrode crushing strength and electrical resistivity.

Acknowledgment

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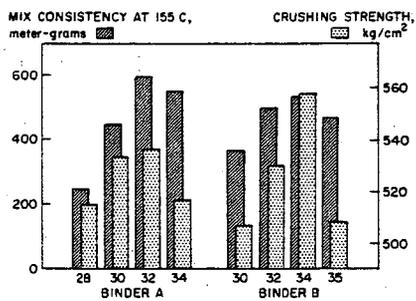
References

1. H. W. Nelson, "Viscosity Stability of Binders for Soderberg Electrodes," paper presented at New York City meeting of American Institute of Mechanical Engineers, February 18-22, 1962.
2. O. Bowitz, T. Eftestol, and R. A. Selvik, "New Methods for Testing Raw Materials for Anode Carbon Paste," paper presented at New York City meeting of American Institute of Mechanical Engineers, February 18-22, 1962.
3. F. A. Smith and A. J. Lombardo, "The Relationship Between the Consistency of the Green Electrode Mix and the Properties of Test Electrodes," paper presented at the New York City meeting of the Fuel Division of the American Chemical Society, September 11-16, 1960.
4. H. L. Jones, Jr., A. W. Simon, and M. H. Wilt, "A Laboratory Evaluation of Pitch Binders Using Compressive Strength of Test Electrodes," Journal of Chemical Engineering Data, 5, No. 1, pp. 84-87 (1960).



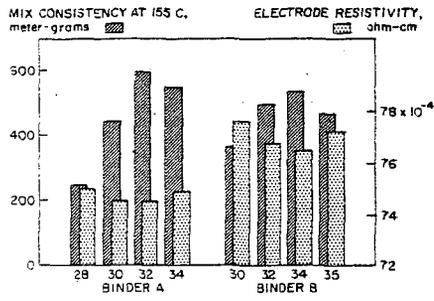
The Brabender Plastograph

Figure 1



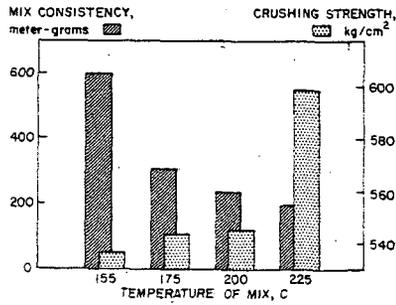
Relationship Between Mix Consistency and
Electrode Crushing Strength

Figure 2



Relationship Between Mix Consistency and Electrode Resistivity

Figure 3



Effect of Mixing Temperature on Mix Consistency and Electrode Crushing Strength

Figure 4

PROPERTIES OF PITCHES

	PITCH A	PITCH B
SOFTENING POINT (CIA), C	109.5	109.0
BENZENE INSOLUBLE, wt %	29.3	34.3
QUINOLINE INSOLUBLE, wt %	10.3	14.3
COKE VALUE (CONRADSON), wt %	58.5	58.3
SPECIFIC GRAVITY 60 F/60 F	1.32	1.29

Table I

PARTICLE-SIZE DISTRIBUTION
OF PETROLEUM-COKE

TYLER MESH SIZE	FRACTION SIZE, weight percent
-0.525 in. +3 MESH	5.0
-3 +4 MESH	5.0
-4 +10 MESH	15.0
-10 +20 MESH	10.0
-20 +48 MESH	15.0
-48 +200 MESH	20.0
-200 MESH	30.0

Table II

EFFECT OF TIME ON MIX CONSISTENCY
AND ELECTRODE PROPERTIES

TEMPERATURE, C	155		225	
	0	7.5	0	24
HOLD TIME, hours				
MIX CONSISTENCY, meter-grams	600	1000	190	600
ELECTRODE STRENGTH, kg/cm ²	536	622	598	642
ELECTRODE RESISTIVITY, ohm-cm x 10 ⁻⁴	74.5	63.2	73.9	63.0

Table III