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EFFECTS OF MIXING VARIABLES AND MOLD TEMPERATURE ON PREBAKED ANODE QUALITY

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Abstract

Bench-scale work was carried out to determine effects of mixing time on quality of pressed and vibrated prebaked anodes. Effects of coke and pitch preheating and mix and mold temperatures were determined also. Green and baked apparent densities of pressed anodes increased by 0.02 Mg/m^3 on increasing mixing time from 5 to 60 minutes. Most of the density increase occurred and baked anode electrical resistivity minimized after 30 minutes of mixing. Coke and pitch preheating had little effect on the bench-scale pressed anodes. A mold temperature 30°C over the pitch softening point (cube-in-air) improved pressed anode properties compared with a mold temperature 10°C over the softening point. Vibrated anode properties were optimum with a 15-minute mixing time. With longer mixing times, properties were greatly degraded. A mix and mold temperature 40°C over the pitch softening point was optimum for vibrated anodes.

Introduction

Although considerable systematic work on effects of raw materials, formulation, and forming parameters on prebaked Hall cell anodes has been reported, little has been published on effects of mixing parameters. Kravtsov, et al. (1), reported minimum anode electrical resistance with 30-40 minutes of mixing, minimum porosity after 30 minutes, and satisfactory strength after 20 minutes, under particular industrial conditions. Effects of coke preheating were described by Zalivnov, et al., (2). This report describes effects of mixing time on bench-scale anode quality, for both pressed and vibration-formed anodes. It includes, also, effects of coke and pitch preheating and mold temperature. It is not anticipated that the results given herein can be applied quantitatively on a commercial scale; however, it is believed that the work can serve as a guide for optimizing commercial fabrication parameters.

Experimental

For these experiments, a medium bulk density calcined coke was used [vibrated bulk density (ASTM Method D4292; -28+48 mesh) = 0.86 Mg/m^3]. Aggregate sizing was a typical plant sizing, but no butts were included. Binder was a typical 110°C softening point (cube-in-air) coal tar pitch. Mixing was carried out in a 3.8 litre (1-gallon) sigma-blade mixer.

Fifty-millimeter diameter by approximately 150 mm long anodes were pressed at 27.6 MPa (4000 psi) or vibration formed. A compressed air-actuated bench-scale vibration former was used. Since varying air pressure and the follower mass were the only means for changing vibration parameters, relatively little optimization of these parameters could be carried out. For the experiments described, follower frequency was 17 Hz and follower mass was 12.7 kg. Compaction time was 3 minutes.

Under the conditions used, 19-wt% pitch was found to be optimum (Figure 1). Green anodes were supported in packing coke and baked under nitrogen at an upheat rate of 25°C/h to a maximum temperature of 1125°C. Green and baked apparent densities and electrical resistivities at room temperature were measured.

In a preliminary experiment to determine rapidity of heat transfer in the mixer, coke at room temperature was added to the mixer preheated at 140°C and temperature measured after 5 minutes. In this time, temperature of the coke increased to 133°C. Because of this rapid heat transfer in the bench-scale mixer, it was obvious that subsequent experiments on mixing time would yield data primarily on interactions between pitch and coke at the final mixing temperature.

A series of experiments on pressed anodes containing 19-wt% pitch were carried out with the following variables:

Aggregate preheat temperature	80, 120, 160°C
Pitch preheat temperature	None, 170°C
Press mold temperature	120, 140°C
Mixing time	5, 10, 20, 30, 45, 60 minutes

Table I gives properties of 66 anodes made from 12 mixes. When pitch was not preheated, visual examination of the mixes after 5 minutes indicated that mixing was incomplete, so no anodes were formed.

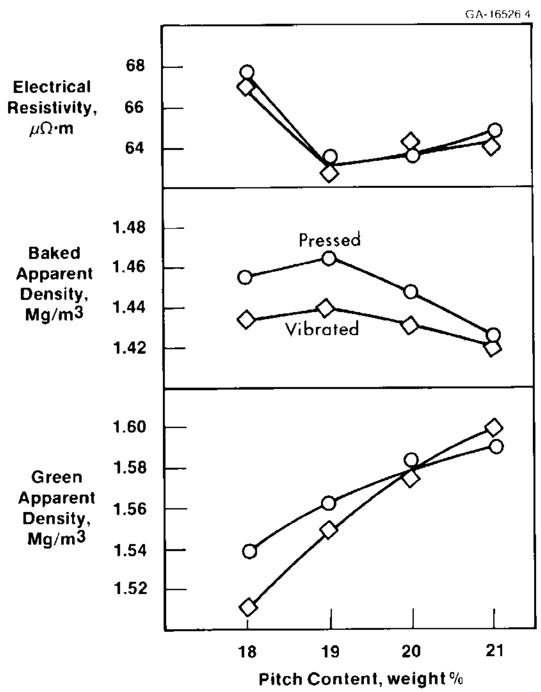

Figure 1 — Effect of pitch content on properties of pressed, (P), and vibrated, (V), anodes.

Table I. Results of Mixing Time Experiments

Temperature Conditions, °C			Mixing Time	Apparent Density Mg/m^3		Electrical Resistivity
Mold	Aggregate Preheat	Pitch Preheat at 170°C	min	Green	Baked	μΩ·m
			5	-	-	--
			10	1.551	1.433	69
120	80	No	20	1.557	2.445	66
			30	1.557	1.454	64
			45	1.561	1.454	63
			60	1.562	1.456	65
			5	-	-	--
			10	1.551	1.443	66
120	120	No	20	1.554	1.451	65
			30	1.554	1.450	64
			45	1.559	1.454	65
			60	1.559	1.464	64
			5	-	-	--
			10	1.557	1.453	66
120	160	No	20	1.549	1.444	64
			30	1.549	1.447	66
			45	1.553	1.454	66
			60	1.558	1.459	66
			5	1.549	1.449	70
			10	1.553	1.454	68
120	80	Yes	20	1.562	1.460	66
			30	1.569	1.466	64
			45	1.573	1.472	64
			60	1.576	1.472	63
			5	1.546	1.438	70
			10	1.548	1.438	67
120	120	170	20	1.553	1.444	67
			30	1.555	1.454	64
			45	1.560	1.454	66
			60	1.556	1.450	68
			5	1.551	1.446	68
			10	1.550	1.448	68
120	160	170	20	1.549	1.454	66
			30	1.555	1.458	64
			45	1.562	1.461	66
			60	1.556	1.460	66

(continued)

Table I. Results of Mixing Time Experiments (continued)

Temperature Conditions, °C			Mixing Time	Apparent Density Mg/m³		Electrical Resistivity
Mold	Aggregate Preheat	Pitch Preheat at 170°C	min	Green	Baked	μΩ·m
			5	-	-	--
			10	1.561	1.453	66
140	80	No	20	1.564	1.460	63
			30	1.567	1.461	63
			45	1.572	1.463	63
			60	1.571	1.472	62
			5	-	-	--
			10	1.556	1.448	66
140	120	No	20	1.570	1.463	63
			30	1.572	1.462	63
			45	1.576	1.466	63
			60	1.579	1.474	63
			5	-	-	--
			10	1.551	1.448	66
140	160	No	20	1.559	1.453	65
			30	1.564	1.453	63
			45	1.570	1.457	65
			60	1.573	1.466	64
			5	1.535	1.441	70
			10	1.546	1.452	67
140	80	170	20	1.551	1.457	66
			30	1.557	1.465	64
			45	1.560	1.472	64
			60	1.569	1.473	64
			5	1.545	1.446	70
			10	1.554	1.454	67
140	120	170	20	1.558	1.463	66
			30	1.562	1.466	64
			45	1.569	1.470	63
			60	1.574	1.475	66
			5	1.541	1.458	68
			10	1.553	1.458	65
140	160	170	20	1.556	1.460	65
			30	1.559	1.462	64
			45	1.567	1.474	64
			60	1.565	1.474	66

Figure 2 shows anode properties as a function of mixing time, averaged for all 12 mixes. Both green and baked apparent densities increased by 0.02 Mg/m³ over the mixing time range examined. About two-thirds of the increase took place during the first 30 minutes. Electrical resistivity did not continue to decrease over the entire range, but minimized after 30 minutes.

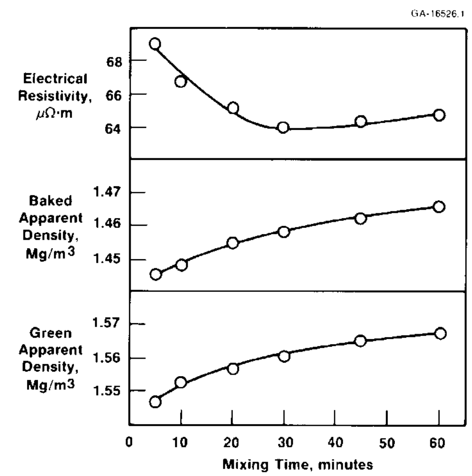

Figure 2. Effect of mixing time on pressed anode properties. (Average for 12 mixes.)

Electrical resistivity may be influenced by the relative amounts of binder coke formed in aggregate coke pores and bridging aggregate particles. The amount of bridging coke may decrease with extended mixing, giving a higher density but poorer electrical contacts among particles.

Based on these results, about a 30-minute mixing time was optimum, even though baked apparent density increased slightly with additional mixing. In a commercial scale mixer with slower heat transfer, additional time would be required with nonpreheated materials.

Aggregate preheat temperature had no apparent effect on anode properties or an optimum mixing time. Thus, the only effect of preheating aggregate is shortening mixing time in a commercial scale mixer with slower heat transfer. The effect of starting with pitch at room temperature or pitch at 170°C was relatively insignificant (Figure 3). Each point in this figure represents an average for 6 mixes. There was no effect on optimum mixing time.

Mold temperature had a small effect on anode properties (Figure 4). Each point in this figure represents an average for 6 mixes. Decreasing mold temperature from 140 to 120°C reduced green and baked apparent densities by 0.01 Mg/m³ and increased electrical resistivity by about 1 μΩ·m.

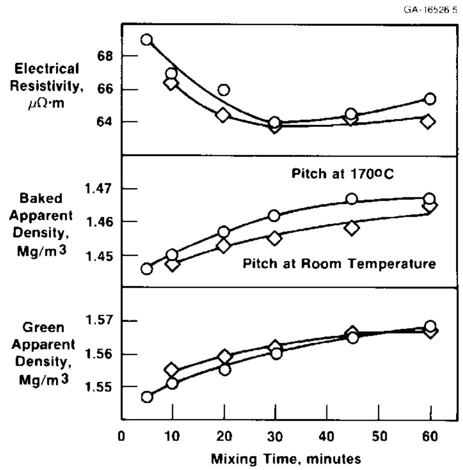

Figure 3 — Effect of pitch preheat on anode properties vs. mixing time relationship. (Average for 6 mixes.)

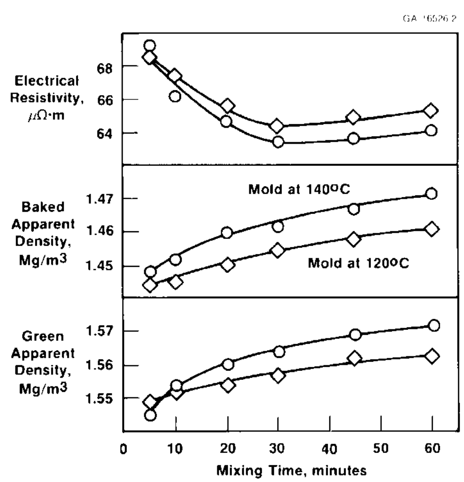

Figure 4 — Effect of press mold temperature on anode properties vs. mixing time relationship. (Average for 6 mixes.)

For vibration-formed anodes, a 150°C mixer and mold temperature was selected, based on the results shown in Figure 5. Aggregate was preheated at 150°C and pitch was at room temperature.

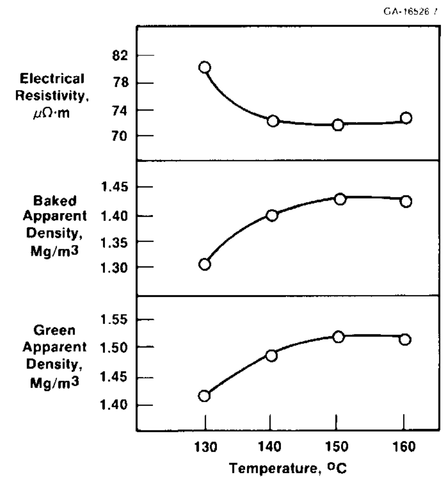

Figure 5 — Effect of mixer and mold temperature on vibrated anode properties.

Figure 6 shows properties as a function of mixing time, averaged for 3 mixes. In contrast to pressed anodes, both green and baked apparent densities and electrical resistivity optimized in about 15 minutes.

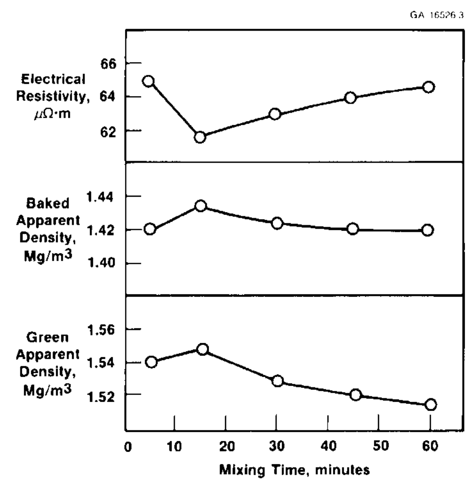

Figure 6 — Effect of mixing time on vibrated anode properties. (Average for 3 mixes.)

To make a more direct comparison between pressed and vibrated anodes, another set of experiments was carried out. From each of several mixes, varying in pitch level, a pressed anode was formed, followed by a vibrated anode, another pressed anode, and one more vibrated anode. Times were not controlled precisely, but the first pressed anode was made after 15 minutes of mixing; each pressed anode took 4 to 5 minutes to produce and each vibrated anode took 7 to 8 minutes. Therefore, fabrication of the second vibrated anode was started after about 30 minutes of mixing.

Results are given in Figure 7. For vibrated anodes, green apparent density decreased with extended mixing, baked apparent density decreased except at high pitch levels, and electrical resistivity tended to increase. In general, pressed anode quality improved with extended mixing over the range covered. These results are in agreement with those shown in Figures 2 and 6.

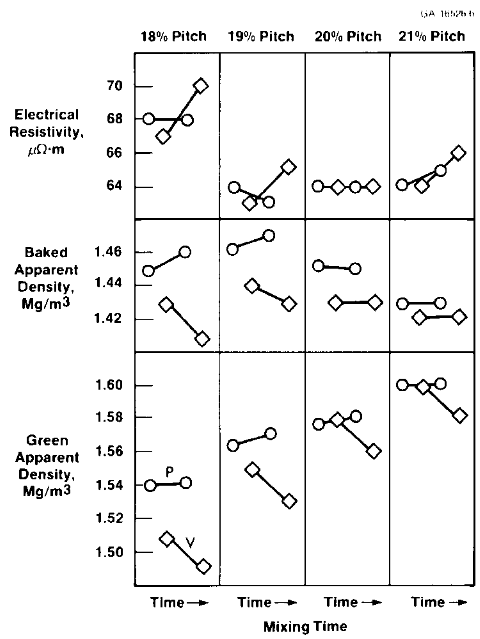

Figure 7 — Effect of mixing time on pressed, (P), and vibrated, (V), anode properties. (Mixing time range is 15 to 30 minutes.)

The reason for the significant degradation of properties of vibrated anodes with increasing mixing time was not determined. It seems obvious, however, that after extended mixing the binder penetrates less deeply into coke pores during vibration. Although this would suggest an increase in binder viscosity, it seems unlikely that the binder itself is changed appreciably by heating for less than an hour at only about 40°C above its softening point. Possibly, viscosity increases due to increased incorporation of coke fines into the pitch with extended mixing.

Conclusions

Anode quality is significantly affected by different time-temperature relationships involving the raw materials, mixer and mold. It is possible to degrade anode properties by overmixing, especially with vibrated anodes. Such effects could be subtle. For example, if mixing time were optimum with given coke and pitch preheat temperatures, increasing these temperatures could be detrimental to anode quality if such a change increased the time that the mix is at the maximum temperature. Other increases in time or temperature beyond those required for optimum properties do not necessarily degrade properties but can waste energy and increase maintenance problems.

References

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2. V. I. Zalivnov, V. N. Mladentsev, and S. I. Sharayda, "Performance of the Petroleum Coke Preheaters Prior to Mixing with Pitch to Make Anode Paste," Tsvet. Met., 11 (1981) pp. 14-15.