THE INFLUENCE OF QUINOLINE INSOLUBLE PITCH CONSTITUENTS ON THE IMPREGNATION BEHAVIOUR

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Introduction

The continual increase in both the electrical and mechanical duties imposed in the electrodes used in arc furnaces has to be matched by improvements in both the raw materials and in the graphite technology. Particularly the mechanical and electrical properties of electrodes can be increased by impregnation of the carbon or graphite artifacts.

The tendency of using binders with higher softening points results in the increase of the bulk density and the decrease of the porosity. Any second impregnation step is rendered difficult by the decreased porosity. In order to improve the efficiency parameters of the impregnation have been studied.

The influence of the impregnation on both the pitch composition and the electrode properties was studied by G.W. Plewin et al. (1). In this work it could be shown that the infiltration of medium and low molecular weight compounds of pitch is favored. The negative influence of the quinoline insolubles has been pointed out but studies on the influence of the quinoline insoluble pitch constituents on the infiltration velocity has not been made.

This paper is concerned with this question and will show that the content of quinoline insolubles is one of the important factors for the selection of pitches.

Experimental

To understand the mechanism of the impregnation behaviour of carbon and graphite three questions have to be answered:

1. Are the quinoline insoluble pitch constituents infiltrated into the artifacts?
2. If the quinoline insoluble pitch constituents can not be infiltrated, which is the influence of the filter cake formed onto the surface on the infiltration velocity of the other pitch constituents?
3. How is the overall impregnation behaviour of pitch in respect to temperature, pressure and especially to QI-content?

The first question can not be answered by the knowledge of the pore size distribution of baked carbon artifacts and of the grain size distribution of the QI. Both the pore size distribution and grain size distribution are within the same range of size, but the pore diameter is normally smaller than the grain diameter. This explains the well known difficulties in the impregnation of carbon artifacts with very small pores using commercial soft pitches the QI of which is larger than 3 %. To study the infiltration behaviour of quinoline insoluble constituents of selected high and low temperature tars and of a special impregnation pitch an filtration experiment was performed with an 5 mm thick disc cut from a carbon electrode. The applied pressure difference was 1 bar.

The influence of a filter cake formed by segregated quinoline insoluble pitch constituents was examined with a porcelain filter disc on which a layer of these constituents of different thickness (0,26 - 2,6 mm) was sedimented. Anthracene oil was used as a model liquid as its viscosity at room temperature is nearly the same as the viscosity of pitch at about 200°C. Furthermore the molecular structure of the anthracene oil is comparable with the structure of the pitch constituents.

The pressure difference was varied between 0,3, 0,6 and 1 bar. The influence of the thickness of quinoline insoluble layers on the porcelain filter disc were measured as the relative filtration rate related to the filtration rate of anthracene oil measured with the clean filter disc.

The overall impregnation behaviour of commercial pitches in respect of temperature, pressure and QI-content was measured with a filter disc made from carbon having a thickness of 5 mm. The filtration pressure could be increased up to the limit given by the mechanical strength of the carbon disc. The examinated temperature range was 180 - 230°C.

Results

The results of the infiltration tests constituents are summarized in Table 1. These results show that especially in a low temperature tar the quinoline insoluble constituents must have a very small grain size. About one third of the QI penetrates the carbon disc, whereas two thirds of the QI are retained at the surface as a filter cake. On the other hand the grain sizes of the QI's of the high temperature tar and the impregnation pitch must be larger than the average pore diameter of the carbon disc. The QI's are almost completely retained. Furthermore the filtration rate is decreased from 13,4 to 7,0 g/h by increasing the QI content from 0,6 to 13 %. The low filtration rate of the impregnation pitch is mainly caused by the larger viscosity of about 100 k Pa s.

To determine the influence of the filter cake containing the quinoline insoluble constituents the filtration rate was measured in a second experiment. The tests showed that the filtration rate of anthracene oil decreases with increasing filtration time. The reason for this strange behaviour is a swelling of the filter cake whereby the quinoline insoluble constituents are forming a kind of colloidal skin (Table 2).

The following experiments were performed with swelled and sedimented quinoline insoluble constituents only. The influence of pressure difference and thickness of the filter cake on the relative filtration rate are shown in Table 3. An increasing pressure difference causes a degressive decrease of the relative filtration rate. An increasing layer thickness results in a nearly linear increase of the relative filtration time at low pressure differences of 0,33 bar, whereas at a pressure difference of 1 bar the relative filtration time is hardly changed if the layer thickness is doubled from 1,3 to 2,6 mm. This means that the filter cake containing QI is compressible. The low relative filtration time at a layer thickness of 0,26 mm can only be explained by an incomplete layer of QI on the disc. It can be calculated that a filter cake of QI up to 2 mm thickness will be formed in an industrial impregnation process using an impregnation pitch with a QI content of 2 - 3 %. The strong influence of the QI content of the impregnation pitches on the filtration rate was confirmed by experiments using different pressures and temperatures.

Reference

1) G.W. Plewin et al., Chimija twerogo topliwa, 6, 95 (1972)

Table 1: Infiltration behaviour of the quinoline insoluble constutuents of a low temperature and a high temperature tar and of a commercial impregnation pitch

	low temperature tar	high temperature tar	impregnation pitch
Softening point (KS) °C	20	20	61
QI content, %	0,6	13	3,5
Temperature °C	120	140	160
Filtration rate g/h	13,4	7,0	2,1
QI content of the filtrate, %	0,25	0,01	0,6
Percentage of penetrated QI, %	30	0	2

Table 2: Unswelled and swelled QI-layers of 1,3 mm thickness. Filtration time of 5 ml anthracene oil.

Filtration cycle	1	2	3	4
Filtration time (min, sec.) of unswelled QI	2'55"	2'40"	3'50"	4'15"
Filtration time (min, sec.) of swelled QI	17'53"	17'30"	17'32"	17'28"

Table 3: Influence of pressure difference and thickness of the QI filter cake on the relative filtration time

Thickness of the filter cake (mm)	0	0,26	0,78	1,3	2,6
Pressure difference (bar)					
0,33	1	6	28	41	87
0,66	1	3	18	25	37
1,00	1	1,5	14	18	20