LABORATORY VACUUM DISTILLATION SIMULATION AND VERIFICATION OF QI INCREASE

B. Gerber and M.C. Botha
Product Technology
Suprachem
South Africa

Introduction

Coal-tar pitches are extensively used as a binder for carbon electrodes in the production of steel and aluminium. The manufacture of a consistent quality pitch for a specific application, depend directly on the following:

- the properties of the feedstock material (crude tar)
- the method and conditions under which the crude tar has been distilled
- the applicant specifications.

The aim of this study is to verify certain property correlation's between crude tar, the distillation process and the final pitch product.

The amount of quinoline insolubles is a critical property and specification for binder pitch applications. There are basically three types of Quinoline Insoluble (QI) materials.[1] Normal (primary, natural) QI which is produced by thermal cracking of the organic components during the manufacture of coke in coke ovens. Carryover QI which is fine coal, char or coke particles entrained in the gases and thus collected with the tar during the coking process. Secondary QI is formed by polymerisation of the aromatic species in pitches subjected to heat treatment temperatures (T~400° C).[2]

The change in the QI content during a step-by-step distillation from crude tar to pitch was verified on laboratory scale to be able to precisely forecast final pitch QI-content. The laboratory distillation was simulated according to the plant operating conditions and specifications.

Experimental

Tars from seven different sources containing a variable amount of QI respectively were used in this study.

The tars were vacuum distilled at reduced pressures ranging from 86 kPaA to 42 kPaA. The vacuum distillations were carried out in laboratory glassware, using an electrical heating mantle. The apparatus was altered so that inprocess samples could be taken from the distilling tar at certain stages of the distillation process, without breaking the vacuum conditions. The apparatus was further adjusted so that the distillates could be collected, while the vacuum conditions were maintained.

The samples and the distillates were collected at four predetermined distillation conditions, according to the plant operating conditions and disitillation process. These conditions were accurately monitored so that the best simulations of the plant distillation process could be obtained on laboratory scale.

The oil and pitch yields were determined gravimetrically. The QI-content was determined in the crude tar, after each distillation step and in the final pitch residue according to ASTM Method D2318-86 (Revised 1991). The softening point of the pitch residues was tested according to STPTC Methods[3] and by ASTM Method D 3104-87 (Reapproved 1991). All seven tars were distilled on laboratory scale and the results obtained after each step compared with the results obtained from the plant also after each step.

Results and Discussion

For illustration purposes two tars have been selected to verify correlation's between laboratory and plant results and conditions. The changes in the QI-content of the crude tars during the laboratory and plant distillation process are presented in Table I. All tars have been distilled to a pitch residue with a comparable softening point. Table II.

Table I QI content after each distillation step

Tar	QI (%) of tar	QI (%) after step 1	QI (%) after step 2	QI (%) after step 3	QI (%) after step 4
Type A Lab	1.77	1.94	2.82	3.34	3.48
Type A Plant	1.77	1.90	2.73	3.21	3.39
Type B Lab	2.69	2.80	3.94	4.55	5.51

Table II Softening Point of pitch residue

Pitch distilled from Tar type	Softening point (° C)
Pitch A- Lab	83.5
Pitch A - Plant	82.4
Pitch B - Lab	84.0

The results in Table I show excellent correlation's between the laboratory and plant results. The results from all the tar sources verified the accuracy of the vacuum distillation simulated on laboratory scale in comparison with the plant operation conditions.

The QI-content and the oil yield after each distillation step are presented in *Figure 1.*

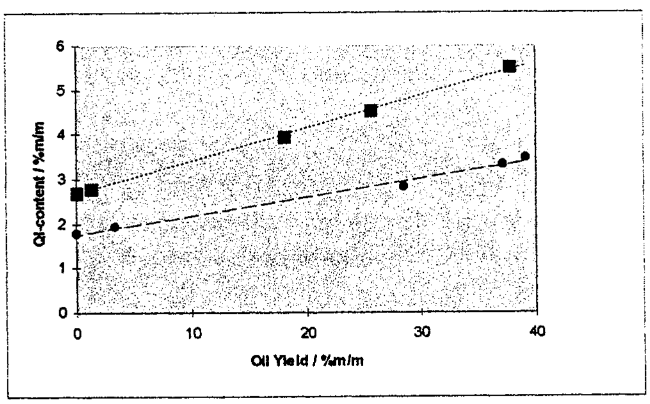

Figure 1. QI-content versus Oil yield for Type A and B tar during laboratory vacuum distillation.

It is clear from *Figure 1* that the increase in QI-content is linear dependant on the pitch yield. Applying linear regression to these curves gives the following equations:

- for type A: $y=0.0419x + 1.7645$ with $R^2 = 0.9891$
- for type B: $y=0.074x + 2.6756$ with $R^2=0.9986$

It is also clear from *Figure 1* that the increase in QI-content during distillation are only due to the concentration effect of the quinoline insoluble materials in the remaining pitch residue under non-polymerisation conditions. No secondary QI was formed in all the residue under non-polymerisation conditions. No secondary QI was formed in all the pitches obtained and this verifies that secondary QI is only formed in distillation processes when high temeperatures (T>400°C) are favourable and taking into account the reactivity of trhe tar source towards the specific temperature process, distillation or heat treatment.[4]

Deviations up to ± 5 % in the pitch and oil yields was found comparing laboratory and plant results and a higher or lower pitch yield directly influences the QI-value. Plant operating problems mainly accounted for these deviations, for example vacuum problems due to leakages on flanges or pump failures.

Conclusion

The QI-content of pitches are influenced by the crude tar feedstock and the conditions under which it has been distilled to form a pitch. The final pitch QI-content can be accurately calculated by knowing the QI-content of the feedstock crude tar and the pitch yield obtained from the tar under a constant vacuum distillation process and at temperatures below polymerisation conditions. This enables one to manufacture a constant in specifications pitch product by accurately controlling the quality of the feedstock material.

Acknowledgement

The author is grateful to all co-workers at Suprachem for their help in the preparation of this paper.