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Short communication

Correlations between quinoline and 1-methyl-2-pyrrolidinone insolubles in petroleum pitches

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Abstract

Quinoline insoluble content (QI) still constitutes one of the most important physico–chemical parameters for evaluating pitches. However, quinoline is highly toxic, expensive, and the ASTM D2318 procedure is very time consuming. The solvent 1-methyl-2-pyrrolidinone (NMP) is less expensive, less toxic, and has recently become preferred as a pitch solvent by many researchers. Nonetheless, its use has not yet become standard for carbon researchers or the carbon industry because it has not been correlated with conventional QI results. The present article establishes correlations between quinoline insolubles (QI) and 1-methyl-2-pyrrolidinone insolubles (NMPI) for petroleum pitches using glass microfiber filters instead of the usual porous crucible.

Keywords: Quinoline; 1-methyl-2-pyrrolidinone; Petroleum pitch

1. Introduction

The two main sources of pitch are coal tar and FCC decant oil from the catalytic cracking of heavy petroleum fractions. Restrictive environmental legislation concerning the emission of toxic and carcinogenic fumes at workplaces and the closing of numerous coke plants in some countries have led to the search for new pitches that would be capable of at least partially replacing coal tar pitches. In this respect, petroleum pitches could provide a good alternative in a market which has been dominated by coal tar pitches [1–4].

Enormous effort has been undertaken to identify the chemical compounds that compose pitches, but the vast amount of hydrocarbons present makes this task almost impossible. Several techniques have been used, with some success, to characterize the original pitch and its chemical changes upon pyrolysis, but they are very time consuming and expensive [5]. The most widely used technique for characterizing a pitch is probably that which concerns its solubility in various solvents [6,7].

Quinoline insoluble content (QI) is a routine parameter used in every carbon laboratory and carbon industry. It is determined by the standard ASTM D 2318 test method, which is a very time consuming procedure, mainly for highly anisotropic pitches [8,9]. Several researchers substitute quinoline by 1-methyl-2-pyrrolidinone (NMP), because this latter solvent is less toxic and less expensive than the former [10–13].

In the present work, QI and NMPI results were correlated with each other, and the porous porcelain crucible, which is the standard filtering device used in the ASTM D 2318 method, was substituted by a glass microfiber filter. The use of glass microfiber filters considerably accelerates this procedure even for highly anisotropic pitches.

2. Experimental

The QI contents of several petroleum pitch samples, produced from three different FCC decant oils, were determined by the standard ASTM D 2318 technique. These

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results were plotted against NMPI values from the same pitches, using the Whatman GF/C glass microfiber filter. Each experiment was repeated at least three times, and all results showed less than two absolute standard deviations from the mean value.

3. Results and discussion

Preliminary results show that the glass microfiber filter and porcelain crucibles have similar abilities for retaining NMPI. However, a large difference in the insolubles retained by either filtering device was observed when the solvent was changed from NMP to quinoline. This difference was found to be higher for QI values below 60%. The experimental results are presented in Table 1.

A straight line was fitted to the data (Fig. 1), and the obtained correlation factor ($R = 0.99656$) suggested that the results could be correlated to each other by the following equation:

$$\mathrm{QI(ASTM\ 2318)} = -7.89847 + 1.07533 \times \mathrm{NMPI(glass\ microfiber\ filter)} \quad (1)$$

It is evident that a negative QI value would be obtained for NMPI values below 7.35%, which means that this correlation would involve some inaccuracy for low NMPI pitches. A set of data from low NMPI samples was therefore analyzed, the results of which are presented in Table 2. A straight line was also fitted to these data (Fig. 2), and the results could be correlated to each other by the following equation:

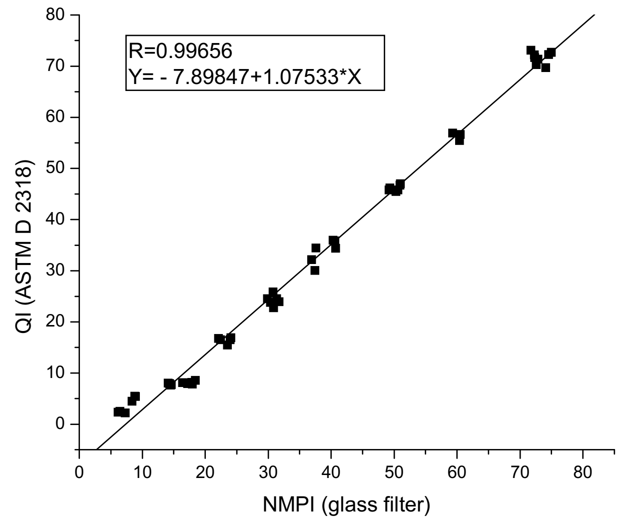

Fig. 1. Correlation between QI values (ASTM D 2318) and NMPI values (glass microfiber filter), from the results of Table 1, for the three pitches PPP, PPB and PPC.

$$\mathrm{QI(ASTM\ D\ 2318)} = 0.1835 + 0.5964 \times \mathrm{NMPI(glass\ microfiber\ filter)}. \quad (2)$$

The results show that Eq. (2) is preferable for converting NMPI, using microfiber glass filter, into QI (ASTM D 2318) when the NMPI values are below 16.87%.

Straight lines would be very convenient for fast and simple calculations, particularly at industry laboratories that only work with pitches that would fall above or below 16.8%. However, a third order polynomial curve (Eq. (3))

Table 1
Results from QI (ASTM D 2318) versus NMPI (glass microfiber filter) in petroleum pitch samples produced from 3 FCC decant oils (PPP, PPB and PPC)

Samples PPP	QI (%)	NMPI (%)	Samples PPB	QI (%)	NMPI (%)	Samples PPC	QI(%)	NMPI (%)
021 A3	7.94	14.33	M 7 A1	–	–	M 12 A1	–	–
*	8.02	14.10	*	5.38	8.92	*	2.48	6.44
*	7.72	14.64	*	5.46	8.83	*	2.21	7.28
*	7.94	14.34	*	4.48	8.36	*	2.33	6.19
*	7.63	14.52	*	–	–	*	–	–
005 A3	16.74	22.13	M 7 A2	–	–	M 12 A3	–	–
*	16.49	22.43	*	7.91	17.18	*	8.09	17.76
*	16.49	23.96	*	8.15	17.83	*	8.57	18.42
*	16.87	24.08	*	8.11	16.40	*	7.83	17.97
*	15.47	23.51	*	–	–	*	–	–
005 A4	34.45	37.61	M 3 A4	24.55	29.89	M 12 A2	22.77	30.86
*	30.6	37.43	*	23.78	30.37	*	23.92	31.72
*	32.16	36.91	*	25.86	30.77	*	24.51	31.34
013 A6	34.37	40.71	M 7 A4	45.80	49.20	M 12 A4	46.70	50.94
*	35.99	40.31	*	46.17	49.34	*	47.04	51.01
*	35.83	40.59	*	45.51	50.31	*	45.80	50.61
005 A6	73.10	71.74	M 7 A5	–	–	M 1 A5	–	–
*	71.69	72.34	*	56.91	59.32	*	69.68	74.10
*	72.22	72.28	*	55.44	60.41	*	72.24	74.53
*	70.31	72.58	*	56.63	60.51	*	72.68	74.97
*	71.35	72.85	*	–	–	*	–	–