

Residual diesel range organics in process waters from laboratory fine coal flotation tests

by J. Morris, R. Sellaro and E. Sarver

Abstract ■ Petro-diesel is a common collector for coal flotation, but the partitioning of diesel range organics (DROs) in process streams has not been widely studied. Preliminary research suggested that under normal dosing schemes total residual DRO contents in process waters should be limited to the sub-ppm water-soluble fraction (WSF). Further, polycyclic aromatic hydrocarbons (PAHs) should partition proportionately with DROs. The purpose of this study was to confirm these findings in laboratory flotation tests. Total DRO and specific PAH contents were measured in the waters associated with the concentrate and tailings streams. The results show that concentrate waters consistently had higher amounts of DROs than tailing waters. Moreover, the DRO contents in both streams generally increased with increasing diesel dosage. In line with preliminary research, DROs appeared to be limited to the WSF, and PAHs tended to partition proportionately with DROs. Under typical operating conditions, it appears that residual DROs should not present an environmental concern, but conditions should be avoided that allow significant overdosing of diesel.

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Introduction

Coal preparation is often necessary to produce clean and efficient thermal coal to meet the demands

of the steam and coking coal markets, which have unique contract constraints. A main requirement of preparation is the removal of ash-bearing minerals, such as shale and pyrite, which are separated from coal particles by a variety of processes depending on particle size. Coarse particle size fractions are processed in systems that use variance in particle density (heavy media vessels, heavy media cyclones or spirals), whereas fine size fractions are processed with flotation, which uses the material's surface properties.

The froth flotation process works by exploiting the difference between the hydrophobic nature of coal particles and the hydrophilic nature of ash-bearing minerals. Essentially, tiny air bubbles are introduced into a slurry of fine coal and ash-bearing particles and water in the flotation cell. The coal particles are naturally hydrophobic and thus attach to the bubbles, floating to the top of the re-

actor where they can be recovered. Conversely, the ash-bearing particles are hydrophilic and are transported to the bottom of the cell through the motion of the fluid. In practice, froth flotation also requires a variety of chemical additives, the fate and transport of which are of increasing environmental interest. At the very least, frother chemicals are needed to strengthen the production of stable, ultrafine bubbles that accumulate the floated coal particles at the top of the cell, and collector chemicals are generally needed to increase the hydrophobicity of the coal particles and thus enhance flotation performance (Laskowski, 2001). The collectors are generally oily substances that coat the coal particles and increase their contact angles with water. Conventional collectors include petroleum products like kerosene, fuel oil and, most commonly, petro-diesel (Vijaya Kumar et al., 2010). "Diesel" in this paper refers to petro-diesel.

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Resumen ■ El petrodiesel es un colector común para la flotación de carbón, pero la partición de compuestos orgánicos de diesel (DRO por sus siglas en inglés) en corrientes de proceso no ha sido ampliamente estudiada. Investigaciones preliminares sugieren que bajo esquemas de dosificación normales los contenidos residuales totales de DRO en aguas de procesos deben estar limitados a la fracción soluble en agua (WSF por sus siglas en inglés) sub-ppm. Es más, los hidrocarburos aromáticos poli-cíclicos (PAH por sus siglas en inglés) debería segmentarse de manera proporcional con los compuestos orgánicos de diesel (DRO). El propósito de este estudio fue confirmar estos resultados en pruebas de flotación realizadas en laboratorio. Se midieron los contenidos totales de DRO Y PAH en aguas asociadas con los flujos o corrientes de concentrados y relaves. Los resultados muestran que las aguas con concentrados tenían de manera consistente mayores cantidades de DRO que las aguas de relaves. Es más, los contenidos de DRO en ambos flujos generalmente se incrementaban con el aumento de la dosis de diesel. De acuerdo con la investigación preliminar, los DRO parecían estar limitados a la fracción soluble en agua (WSF) y los PAH tendían a segmentarse proporcionalmente con los DRO. Bajo condiciones de operación típicas, los DRO residuales no deberían representar una preocupación medioambiental, pero deben evitarse situaciones que permitan una sobredosis significativa de diesel.

Diesel is produced as a fractional distillate of crude oil and is a complex mixture of compounds spanning a range of roughly C10 to C19 hydrocarbons, varying not only with the crude oil source but also with the refining processes (Agency for Toxic Substances and Disease Registry, 1999). Because of the complexity and variability of diesel, characterization is difficult. However, broad categories are typically assigned to the diesel range organic (DRO) compounds (Agency for Toxic Substances and Disease Registry, 1995). Saturated hydrocarbons (such as alkanes and cycloalkenes) usually make up about 90 percent of diesel by weight, whereas aromatics (such as naphthalenes, acenaphthenes and acenaphthylenes) make up about 10 percent (Wang et al., 2003).

Since water is used as a continuous phase for all particulate separations within the coal preparation plant and is generally recycled to the extent possible, it may provide a mode of transport for any residual processing chemicals to the environment, or concentrate them within the plant. Relatively small volumes or concentrations of saturated hydrocarbons are not typically considered significant environmental concerns, as they tend to break down easily and generally have low toxicity, and there are currently no federal water quality standards for DROs. However, the aromatics – specifically, polycyclic aromatic hydrocarbons (PAHs) – may be harmful even in small volumes or concentrations. They have thus been given a great deal of attention by research and regulatory communities over the past several decades (Cerniglia, 1993), and many states, such as Maine (Maine Department of Environmental Protection, 2010), are transitioning away from risk assessment and regulatory efforts based on total DROs and moving toward efforts based on specific hydrocarbon groups of concern like PAHs. In addition to being potentially toxic, PAHs are also known to be relatively difficult and/or slow to degrade. Degradation rates vary from a few hours to days in air to several months to years in soil (Scientific Committee on Food, 2002). Diesel is known to contain mostly lightweight PAHs (such as naphthalenes, fluorenes and phenanthrenes) that are typically more volatile and soluble than the higher-molecular-weight PAHs,

as well as easier to degrade (Wick et al., 2011).

Considering potential concerns associated with the environmental fate of DROs, it is prudent to investigate the partitioning of these compounds between various streams from fine coal flotation circuits. Prior to the laboratory-scale froth flotation experiments presented in this paper, we completed other tests concerning DROs. These preliminary tests involved only controlled mixing of fine coal particles with water and diesel, followed by filtration of the water and measurement of total residual DRO and specific PAH contents. Results are reported in detail elsewhere (Morris et al., 2012, 2013), but the main findings are summarized below:

- Even at relatively high diesel dosages in the context of practical operations, for example, 1,000 g/t, raw coal particles had the capacity to sorb most of the diesel. Indeed, results consistently showed that only very small amounts of total DROs partitioned to the water, with sub-ppm concentrations measured for all tests. In general, it was concluded that the measured DROs were largely in the water-soluble fraction (WSF).
- Of the U.S. Environmental Protection Agency priority PAHs (Environmental Protection Agency, 2008) that could be detected in the diesel tested, the PAHs generally appeared to partition proportionately with the total DROs.
- Residual DROs, including PAHs, were removed from water through volatilization under the conditions of the water being exposed to atmosphere along with heating, mixing or aeration.
- When coal particles previously contacted with diesel are rinsed with clean water, DROs can desorb. During initial rinses, excess diesel was removed from the particles, but with successive rinses desorption was limited to the WSF.

To simulate more realistic conditions, we conducted laboratory-scale flotation tests and present the results here. For all tests, the residual total DRO content and

contents of PAHs of interest were determined in the process waters associated with the concentrate and tailings streams. The traditional flotation performance metrics of combustible coal recovery and ash rejection were determined as well.

Experimental methods

A series of flotation tests were conducted using a wide range of diesel dosages on three different coal samples to determine the contents of DROs and PAHs in the concentrate and reject (tailings) streams. Coal samples 1 and 2 were direct flotation feeds, while coal sample 3 was run-of-mine material. The two coal preparation plants, from which the flotation feed samples were obtained, blend from multiple coal seams: Pocahontas No. 3, Sewell and Beckley for coal sample 1; and Red Ash, Lower War Eagle and Sewell for coal sample 2. Coal sample 3 was from the Hernshaw seam, and upon collecting the material it was stored in a sealed barrel lined with plastic to prevent oxidation before crushing and sizing. All three samples were bituminous, high-rank coals with energy densities between 13,000 and 14,000 Btu/lb (30,238 and 32,564 kJ/kg). Coal samples 1 and 2 were prepared for testing only by sizing while coal sample 3 required crushing and sizing, which were performed on the same day. The sized materials were promptly stored in airtight plastic bags and refrigerated to minimize oxidation prior to performing the flotation tests. Coal sample 3 was crushed using a laboratory jaw crusher followed by a roll crusher, and then wet-screened to obtain material in the range of 44 to 149 microns (100 x 325 mesh). Coal samples 1 and 2 were wet-screened at +44 microns (+325 mesh). Following screening, the ash contents of coal samples 1, 2 and 3 were 10.8, 10.6 and 45.7 percent, respectively.

Figure 1 shows the particle size distributions for the three samples that were used in this experiment. For each coal sample, the distribution was determined from five samples and the figure shows particle size by volume – a cumulative percentage – passing through a size-selective device.

The same diesel was used for all tests and was purchased from a local fuel station and stored in an amber, airtight glass jar. The frother used for the tests was a sample of 99+ percent methyl isobutyl carbinol (MIBC), which was purchased from Fisher Scientific (Hampton, NH).

Flotation tests. The laboratory flotation tests were performed in a Denver cell with 2.0 L of deionized water. Although deionized water is not encountered in industrial settings, using the deionized water in these tests eliminated the influence of variable water chemistry on experimental results. In total, 19 tests were carried out including four duplicates for coal sample 2 (Table 1). The MIBC dosage for all tests was 10 ppm, while diesel dosage was varied at 0, 50, 250, 500 and 1,000 g/t, with all dosages being tested on all three coal samples. The slurry's percentage of solids remained constant at 5 percent (by weight). During the flotation tests, the speed was adjusted to 1,300 rpm and the slurry was mixed for two minutes to ensure homogeneity. Diesel was then added at

a predetermined dosage for each test, mixing was carried out for another two minutes, and the frother was added just after that. Next, the air valve was opened completely and, once the froth zone was formed, the concentrate was paddled out for two minutes while the tailings remained in the cell. Both the concentrate and the tailings samples were filtered by vacuum using a 5-micron filter and dried and analyzed for ash content determination while the filtrate was collected for residual DRO analyses. Following dewatering of the concentrate and tailings, the solids for each sample were oven-dried until no incremental weight change could be measured (that is, all moisture was removed). The dewatering equipment, including the vessel and tubing, were thoroughly cleaned between each sample by rinsing with deionized water and methanol. Our prior work showed that the filtration process did not affect residual DRO values when compared with centrifuging to remove particles (Morris et al., 2012).

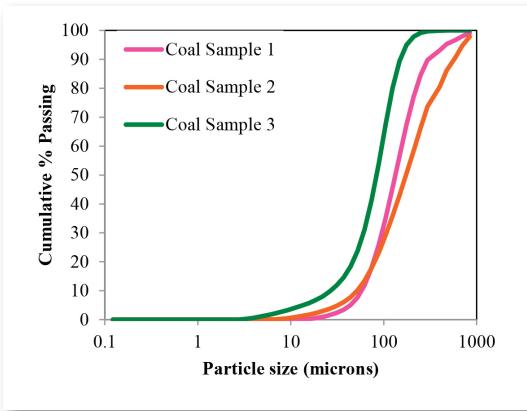
Analytical methods. Total DRO content was determined using an Agilent 5890 gas chromatograph equipped with a flame ionization detector (GC-FID), following EPA Method 3150. Samples were also analyzed for specific PAHs using a Thermo Trace gas chromatograph equipped with a Thermo DSQ II mass spectrometer (GC-MS), following EPA Method 3535A: Solid-Phase Extraction (SPE). The targeted PAHs were chosen based on the results of preliminary tests as described below. Ash content was analyzed in each solid sample using a LECO model 601-400-600 ash analyzer, and the yield and combustible material recovery for each flotation test were then calculated.

Results

Table 1 summarizes the results of the total residual DRO measurements in the concentrate and tailings filtrate streams for each test. (Since some duplicate tests were conducted on coal sample 2, results for this coal sample are displayed as Experiment 1 and Experiment 2.) As expected, the results of the flotation tests consistently indicated that a small fraction of diesel partitioned to the concentrate and

Figure 1

Particle size distributions (by volume) of coal samples 1-3.



Technical Papers

Table 1

Flotation test results.

Test name	Yield (%)	Recovery (%)	Ash rejected (%)	DROs (ppm)	Processing stream
Coal sample 1 0 g/t	92.7	99.2	61.1	N/A	Concentrate
				N/A	Tailings
Coal sample 1 50 g/t	93.0	99.2	58.0	0.12	Concentrate
				0.01	Tailings
Coal sample 1 250 g/t	93.6	99.5	55.2	0.23	Concentrate
				0.07	Tailings
Coal sample 1 500 g/t	93.8	99.5	53.1	0.51	Concentrate
				0.14	Tailings
Coal sample 1 1,000 g/t	93.6	99.4	54.6	0.60	Concentrate
				0.43	Tailings
Coal sample 2 0 g/t	93.1	99.0	56.5	N/A	Concentrate
				N/A	Tailings
Coal sample 2 (Expt. 1) 50 g/t	92.1	98.8	56.5	0.21	Concentrate
				0.01	Tailings
Coal sample 2 (Expt. 1) 250 g/t	92.9	99.0	51.0	0.32	Concentrate
				0.04	Tailings
Coal sample 2 (Expt. 1) 500 g/t	95.6	99.3	31.4	0.30	Concentrate
				0.07	Tailings
Coal sample 2 (Expt. 1) 1,000 g/t	89.2	95.9	59.6	0.45	Concentrate
				0.28	Tailings
Coal sample 2 (Expt. 2) 50 g/t	94.6	99.3	44.2	0.12	Concentrate
				0.00	Tailings
Coal sample 2 (Expt. 2) 250 g/t	93.9	99.1	50.3	0.07	Concentrate
				0.00	Tailings
Coal sample 2 (Expt. 2) 500 g/t	93.1	98.9	55.6	0.11	Concentrate
				0.02	Tailings
Coal sample 2 (Expt. 2) 1,000 g/t	92.5	97.5	50.0	0.55	Concentrate
				0.23	Tailings
Coal sample 3 0 g/t	55.8	87.9	82.4	N/A	Concentrate
				N/A	Tailings
Coal sample 3 50 g/t	63.1	93.5	73.1	0.14	Concentrate
				0.01	Tailings
Coal sample 3 250 g/t	62.0	93.1	74.9	0.01	Concentrate
				0.00	Tailings
Coal sample 3 500 g/t	67.1	94.3	65.3	0.12	Concentrate
				0.02	Tailings
Coal sample 3 1,000 g/t	71.2	94.8	57.0	0.20	Concentrate
				0.03	Tailings

*N/A = Not applicable.

tailings streams, but the total DRO content again appeared to be limited to the WSF of the diesel.

Figures 2-4 show the residual total DRO contents in the concentrate and tailings streams for the three coal samples. For all three coal samples, the residual DRO contents in the concentrate stream samples were significantly higher than those in the tailings streams – generally by a factor of two or more. Moreover, residual DRO contents in the streams tended to increase with increasing diesel dosage. The duplicate tests presented in Table 1 for coal sample 2 show some variation in results, which could be attributed to slight changes in sensitivity of the GC-FID between Experiments 1 and 2. Experiment 1 tests were conducted and DRO contents were measured in the water associated with the concentrate or tailings streams at the same time as all tests on coal samples 1 and 3, but Experiment 2 tests were conducted several weeks later, and oxidation of the coal in the meantime may have caused changes in water chemistry. Although there are some differences between results in Experiments 1 and 2, the trends are consistent for all samples and show an increase in residual DRO with increased dosage. Further, within each experiment for coal sample 2, DRO values for the 50, 250 and 500 g/t diesel dosages tend to be quite similar (including nearly negligible values for all tailings streams), while the DRO values for the 1,000 g/t diesel condition are significantly higher in both the concentrate and tailings streams.

For coal samples 1 and 2, the DRO amounts in the tailings water are greater than those for coal sample 3. It is likely that the observed result is an artifact of the particle size distributions for the coal samples tested. As shown in Fig. 1, coal samples 1 and 2 had significantly larger particles than coal sample 3, and since diesel sorption to the coal particle is surface-area dependent (smaller particles can sorb more diesel), higher residual DRO contents in the concentrate and tailing streams associated with coal samples 1 and 2 should indeed be expected. Further, as diesel dosage is increased for these coals, an “overdosing” situation occurs wherein more DROs report to the tailings. The overdosing that occurs for coal sample 3 is much less pronounced, even at the highest diesel dosage tested.

Prior experimental work showed that naphthalenes make up the largest group of PAHs in the test diesel, and they are also expected to be present in relatively higher concentrations than other PAHs in the flotation process waters (Morris et al., 2013). The GC-MS was used to screen for naphthalene and 22 of its methylated compounds in all the concentrate and tailings water samples. Phenanthrene was found to be present in the test diesel but had not been detected in the water samples during prior preliminary testing. The GC-MS was used to screen for this compound in all water samples resulting from the flotation tests, but again it could not be detected.

In total, six naphthalene compounds were detected: naphthalene, 2-methylnaphthalene, naphthalene C2a, naphthalene C3a, naphthalene C3c and naphthalene C4a. Results are shown here only for naphthalene (Fig. 5) and 2-methylnaphthalene (Fig. 6) since most of the samples contained at least a trace amount of these compounds,

Figure 2

Residual DRO contents in process waters for coal sample 1.

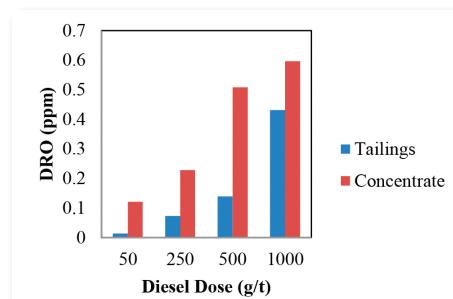


Figure 3

Residual DRO contents in process waters for coal sample 2 (Experiment 1).

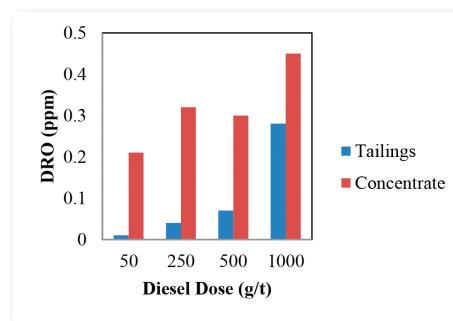
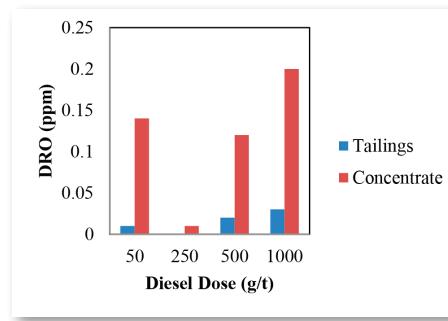


Figure 4

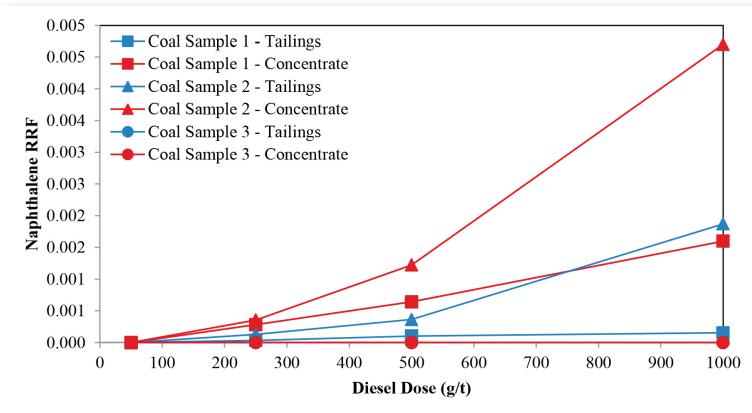
Residual DRO contents in process waters for coal sample 3.



whereas the other four named compounds that were detected were only found in a few samples and at levels that were 10 to 100 times lower (Morris, 2013). Note that results are displayed based on the relative response factors (*RRFs*) derived from sample chromatograms, rather than as calculated concentrations. This is because all results were significantly – 10 to 500 times – lower than the lowest PAH standard analyzed (a 1-ppm solution containing multiple PAHs). Reporting the *RRF* illustrates the relative differences between samples but does not attempt to quantify the PAH concentrations. For some samples, these PAHs appeared to be present in content levels below the GC-MS detection limit, and thus their *RRFs*

Figure 5

Naphthalene in flotation samples (results for coal sample 2 are only shown for Experiment 1).



are shown as a 0.000 value in Figs. 5 and 6. In general, the PAHs appear to be present at higher concentrations in the concentrate samples than in the tailings samples, which is consistent with the trends of total DRO concentrations. Equation (1) shows how the *RRF* is calculated using the sample area, *SA*, and the internal standard area, *ISA*, derived from sample chromatograms:

$$RRF = \frac{SA}{ISA} \quad (1)$$

Discussion

Based on the results of prior preliminary testing (Morris et al., 2012, 2013), it was anticipated that total residual DRO contents would be limited in the flotation process waters associated with the concentrate and tailings, perhaps to the WSF, and the results of flotation tests described here confirmed this expectation. Further, these results also showed that the concentrate process water tends to have higher DRO concentrations than the tailings process water, which is also consistent with prior testing on diesel desorption from coal surfaces (Morris et al., 2013). This phenomenon is likely explained by the fact that diesel sorbs to the coal surfaces but also tends to agglomerate and collect within the froth, hence reporting to the concentrate product. Some of this diesel may be easily removed from the coal during dewatering, which would explain the current results, as well as those of the initial coal “rinses” in prior tests. The flotation test results also confirmed earlier indications that residual PAHs in process waters likely partition proportionately with the total DRO content.

In terms of conventional evaluation of flotation performance, diesel dosage is typically selected to optimize recovery and clean coal ash simultaneously. This dosage may vary substantially based on the coal sample and operational parameters. It should be noted that the two-minute retention time used in the flotation tests in this study was likely too long (the tests were run until exhaustion). While diesel fuel – or other collector – is practically used to enhance the flotation rate of fine coal, this effect is not well demonstrated here. The fact that the addition

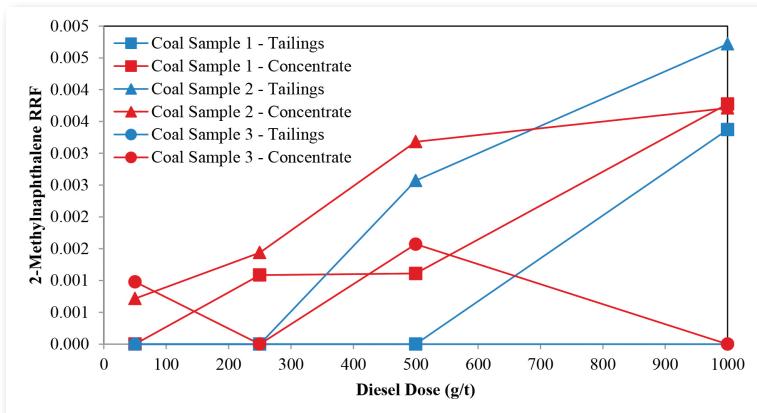
of diesel had little effect on coal recovery in some experiments does not necessarily mean that the flotation rate was not enhanced but rather that the enhancement could not be observed (because it occurred well before the samples were collected). For future investigations, it is recommended that the flotation tests be run over a range of retention times to demonstrate what, if any, differences in diesel partitioning may occur.

Generally speaking, it appears that environmental performance may also benefit from careful diesel dosing, since overdosing clearly tends to allow more residual DROs to unnecessarily partition to concentrate and tailings waters rather than the coal product. In practice, the consequences of overdosing may be substantial since excess diesel tends to depress the froth zone, which limits coal collection and recovery and instead results in more coal – and diesel – reporting to the tailings stream. Thus, options for maintaining proper diesel – and other chemical – dosages should be considered. For example, a programmable logic controller that specifically adjusts chemical dosages based on coal feed characteristics, such as feed rate and ash content, may be particularly attractive, although this may be difficult for plants processing several different coal types with varying product constraints. Of course, any improvements in both operating and environmental performance may ultimately provide economic benefits too.

It should be noted that for the experiments reported here, only a limited range of diesel dosages was explored for a limited selection of coal samples, and within limited particle size distributions. All three coals used here ultimately required relatively low levels of diesel for favorable flotation performance under the experimental conditions, but other coals may have significantly greater collector requirements. Experiments that explore a wider range of typical coal samples, diesel dosage and operating conditions, such as retention time, would give better insights into the possible partitioning of DROs to process waters in the field, especially at the very high end of the diesel dosage range. Moreover, the effect of water chemistry was not explored in this study. Given that deionized

Figure 6

2-Methylnaphthalene in flotation samples (results for coal sample 2 are only shown for Experiment 1).



water (pH of 5.5-6.0) was used in these tests, increased interaction between the aluminosilicate (clay) mineral surfaces and coal might have occurred, which in turn could have reduced diesel sorption onto the coal surface (by competing for sorption sites). Considering that pH – and other water quality parameters – may often change in a plant setting, for example, with changes in the degree of feed coal oxidation that may additionally affect the coal particle hydrophobicity and thus diesel sorption capacity, further investigations of water chemistry effects are needed.

Also noteworthy is the fact that alternative collector chemicals are currently available or in development for fine coal flotation. These include bio-derived and biodegradable products, which appear to be quickly gaining interest. Limited information regarding the production and performance of these chemicals is already available (Eraydin et al., 2012; Hines et al., 2011), and future research efforts and field trials may well result in widespread acceptance by industry.

Conclusion

Considering the collective results presented here and in prior studies by the authors, it appears that under normal operating conditions for fine coal flotation circuits, residual DROs should be present at only very low concentrations. However, should conditions exist where diesel is effectively overdosed, for example, in cases where the flotation coal feed is reduced or halted while diesel continues to be fed into the flotation system, DRO levels in concentrate and tailings waters might be significantly increased. It is therefore recommended to avoid such conditions. Moreover, conscientious diesel dosing should be beneficial since it should increase the performance of the processing system. ■

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