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Analysis of EMR oil-sands samples

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Subject Analysis of EMR Oil-Sands Samples
Sujet

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In the recovery of bitumen from oil-sands by the Hot Water Process the primary product is a froth containing about 60% water and solids. Before the bitumen in this froth can be upgraded it must be treated to remove the bulk of the contaminants. Conventional practise is to dilute the froth with naphtha and then subject it to a sequence of centrifugation and filtration steps to remove the water and solids. This treatment is both complex and costly.

An alternative processing scheme, proposed recently, utilises an RTR solvent extractor-separator as a pretreatment for the froth during naphtha dilution. This scheme has the advantage of providing a more uniform feed to the chosen finishing step be it a centrifuge or inclined settler for example. A series of

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tests have been carried out at the mineral processing branch of EMR to determine the range of operating conditions for a bitumen-naphtha contactor. As part of a collaborative agreement the samples produced during this work were analysed in the Colloids and Clathrates section of the Chemistry Division.

Samples

Samples were taken from the organic and aqueous streams of the contactor both before and after settling. In some tests the middlings emulsion from the settler interface was also sampled. For all runs samples of each stream plus the feed were taken at 3 and 4 hrs. after start-up. A total of 109 different samples were received. It was required to analyse each sample for bitumen, naphtha, solids and water. For each individual analysis it was necessary to sub-sample and this led to increased errors because of the heterogeneous nature of the bulk mixtures. In the absence of a better method a sample (150-100g) was removed quickly from the bulk after vigorous agitation. For the organic based samples sub-sampling resulted in rather poor reproducibility for the water and solids content. The feed samples presented a particular problem in that the high viscosity of the undiluted bitumen prevented any attempt at homogenisation before sampling. In these cases as much of the free water as possible was decanted from the oil and the two phases analysed separately. The results from the two sets of data were then recombined to give an overall analysis for each feed sample.

Experimental

Methods

Organic Phase. The methods adopted were

1) volumetric determination of water in a Dean and Stark Separator, using toluene as the reflux medium 2) colorimetric determination of bitumen by comparison with previously determined standards 3) separation of solids by centrifuging and then ashing the residue to remove associated hydrocarbons 4) determining the naphtha content by difference.

A sample (50-100g) was transferred quantitatively into a 1ℓ round bottomed flask, toluene (300 ml) was added and the mixture refluxed until the volume of water collected in a Dean and Stark trap was constant.

The water and toluene in the side arm of the trap were warmed (50-70°C) to facilitate separation of the phases and the volume of water and temperature noted. The measured volume of water was converted to weight using density values from the Handbook of Physics and Chemistry. Weight percent water in the original sample could then be determined.

The toluene solution remaining in the flask was allowed to cool, weighed, and the supernatant liquid syphoned off into a centrifuge bottle. The liquid phase was centrifuged until a cohesive cake of solid formed, allowing the cleaned liquid to be separated by decantation. The solids from the round-bottomed flask and the centrifuge bottle were quantitatively transferred to

a weighed crucible, solvent was removed by evaporation at 110°C followed by ashing to constant weight at 450°C. Hence the ash content of the original sample was determined.

A portion of the centrifuged toluene solution was taken and diluted with fresh toluene to allow a determination of light transmittance at 530 nm, using a Bausch and Lomb spectronic 20 colorimeter. Duplicate solutions were prepared for each determination. To determine actual bitumen concentrations these results were compared with a calibration curve obtained from a 'standard' sample of bitumen extracted from the original froth material. To prepare the 'standard' a sample of froth was extracted with toluene, centrifuged to separate the solids and then heated under vacuum to remove toluene. The residual toluene content, as determined by NMR, was negligible. A plot of light transmittance vs. bitumen concentration is shown in Figure 1. From the weight of toluene solution and the measured bitumen concentration the amount of bitumen in the original sample could be determined. The naphtha content was then estimated by the difference between the sum of the percentages of bitumen, water and solid contents and 100%.

As mentioned earlier the feed samples were analysed in two parts. The bitumen fraction was treated in the same way as the organic phase, described above, except that in the absence of naphtha each component could be determined directly. The separated aqueous phase was weighed and then evaporated to

dryness to determine the solids. In some cases it was not possible to separate the oil completely from the water and a small amount remained with the solids on drying, however, this was never a very large quantity. A few samples of dried solids were ashed in an attempt to estimate the residual oil. This was not particularly satisfactory as the oil-free solids themselves show a significant weight loss when heated to 450°C. For all the feed samples analysed the average total for all components came to 101.4±1.0%, which was considered to be adequate for the experimental requirements.

The settler interface emulsion material ('crud') was analysed in the same way as the organic phase. Further separation of the organic and aqueous components had occurred during storage, these samples were not reshaken. A sample was withdrawn directly from the interface layer while attempting to avoid sampling the cleanly separated solvent and water phases. Two of these samples, one water rich and one organic rich, were analysed for vanadium content.

Aqueous Phase: 50-100 grams of aqueous tailings were transferred into a preweighed glass jar. 100 ml of carbon tetrachloride was introduced into the sample jar and the jar agitated for 15 minutes to extract the bitumen and naphtha. The contents were allowed to settle until two distinct layers formed.

Determination of the bitumen content: The upper layer of the

contents of the jar, consisting mostly of water, was transferred to a fine pore glass frit filtering funnel until a hole appeared in the clay layer on the interface. An aliquot of the solution of bitumen and naphtha in carbon tetrachloride was then removed and filtered through a small fine pore glass frit filtering tube. A portion of this filtrate was used for bitumen determination by the colorimetric ⁽¹⁾ method. Another portion was used to determine naphtha using a quantitative proton NMR technique ⁽²⁾.

Solids Determination:- after the aliquot of sample extract solution was removed for bitumen and diluent analyses the remaining extract was also transferred to the filtering funnel, along with the residue in the small filtering tubes. The contents were washed several times, first with toluene and then with acetone. The funnel and solids were dried in an oven at 110°C, allowed to cool and then reweighed.

Water determination:- water content of the aqueous tailings was determined by difference, by subtracting the amounts of bitumen, naphtha and solids from the total weights.