APPLICATION OF THE PHASE-EXCHANGE METHOD TO THE DEMINERALISATION OF ATHABASCA BITUMEN-SAND

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1. Introduction

The method of phase-exchange has been successfully applied to the demineralisation of graphite.[1,2] The applicability of the same method for demineralising coal has also been reported and is utilised by the so-called "Convertol Process".[3,4] In these systems, the starting materials are uniformly solids only, carbonaceous material and minerals, physically mixed, and two non-miscible liquids, water and oil are subsequently added. Equilibrium conditions of such systems have been investigated in detail.[1,5] Raw graphite, containing nearly 50% carbonaceous material and 50% mineral impurities or more, is milled in the presence of water into a paste or slurry. To this intimate mixture a mineral lubricating oil is added in a kneading apparatus. The mixture of the four components, however, is not stable, and the system separates into two liquid phases; water and minerals forming one phase, while the oil and graphite form the other phase, resulting in the separation of the solids. The mechanical separation of the two phases is very easy in that the water-mineral phase can be washed off the oil-graphite phase and thus the mechanical separation of the two solid phases is achieved. Unlike the flotation method, always only two clear-cut fractions are obtained.

In these studies it was found that there is a limiting value up to which minerals go into the water to form the water-mineral phase and the rest of the minerals remain in the oil-graphite phase. There is also another limiting value for the water which remains in the oil-graphite phase, while practically no oil remains in the water-mineral phase. The limiting mineral value depends upon the extent to which physical separation has taken place earlier by milling. It further depends on the quantity of the oil added to the system and on the effectiveness of the external shearing forces which are applied to bring about the separation either by stirring or by kneading action.

The method of phase-exchange utilises the interfacial forces of the surfaces of different solids in the presence of two or more immiscible liquids. Application of external mechanical forces, like shearing, helps in bringing about intimate contact between different components and their surfaces, thus enabling a straight separation and high enrichment of the two different solids in their respective liquid phases.

2. Object of Investigation

The present work has been taken up to study the applicability of this principle to such systems, where the four components exist in initial mixtures different from the above mentioned materials. We have chosen systems in which a solid and a liquid already occur as a natural mixture and to which is then added another solid and liquid mixture which also may occur naturally. The affinity of the particular solid for the liquid with which it is naturally found should be less than for the liquid which is being mixed with it later. In the present work naturally occurring Athabasca bitumen-sand[6] (Alberta, Canada) containing approximately 85% sand and 15% bitumen, has been taken as one of the starting materials. To this we have then added (i) peat, containing approximately 80% water and 20% carbonaceous material, (ii) naturally occurring graphite, containing 40–60% minerals and, (iii) wood charcoal, containing about 5% mineral impurities. The extent to which the principle of phase exchange applies to the demineralising of the bitumen-sand and the graphite or coal on one side and to which extent water could be removed from peat on the other side, has been studied.

3. Raw Materials

Athabasca bitumen-sand.[6]—This sand is found as a series of outcrops along over a hundred miles of the Athabasca river and its tributaries in Alberta, Canada. The extent of the country that is underlain by the bituminous sand formation is not fully known but recent studies have shown an area up to 30,000 square miles. The theories regarding the origin of this vast oil sand deposit are not definite. It is calculated that nearly one hundred million barrels of viscous bitumen per square mile may be available.

The mineral aggregates consist mainly of quartzy particles of 100–200 (Tyler standard) mesh size, but other valuable minerals like vanadium and aluminium are also present. The specific gravity (1·027–1·005) and also the viscosity of the bitumen vary to a great extent throughout the deposits from north to south.

From specimens it can be seen that bitumen surrounds the sand grains, covering the surface of the sand particles completely. Separation of the bitumen from the sand particles although not very satisfactory, was found possible by the so-called "Hot water method".[6] Due to a rather high viscosity-temperature gradient of the bitumen it becomes easily liquefied at somewhat higher temperatures and in the presence of water floats away from the sand.

The content of sulfur in the raw bitumen is rather high and varies from 4·5–5·5% by weight of raw bitumen. When the bitumen is distilled, the sulfur is found to be uniformly distributed throughout the whole range of the distillates.

Peat.—The raw peat used in the following experiments was a well humidified sphagnum peat with a water content of 79% and low ash content. The samples were received from Messrs. Stockaryd, Nya Torv A. B. Stockaryd, Sweden. The original water content of the fresh peat was about 90%.

Graphite.—We have used raw graphite obtained from Patna State Graphite Collieries, Titilagarh, with an ash content of 40–65% depending upon the particle sizes. The size fractions which we have used in our investigations are of (− 325) mesh and (− 100 to + 150) mesh (Tyler Standard) sizes. They have ash contents of 51·8% and 43·0% respectively. The ash from the original graphite was analysed and was found to contain about 73·0% silicious material and about 20·0% iron and aluminium.

Charcoal.—The charcoal used in our experiments had an average ash content of 5·0% in (− 150) mesh (Tyler standard) sizes. It is a normal grade wood charcoal.

4. EXPERIMENTAL PROCEDURE

In all our experiments with charcoal or graphite, these substances were first put in the kneader and mixed with a known quantity of water. This mixture was then kneaded for sometime to get a uniform paste. In the case of peat, further mixing with water was not necessary, since it already formed a paste. To these pastes a calculated amount of bitumen-sand was then added slowly. The new mixture was then kneaded thoroughly till the exchange of phases started, as could be observed by the appearance of muddy water at the edges of the apparatus. The kneading was continued further till not only had the exchange of phases been accomplished but also a clear-cut separation was obtained and easy washing of the minerals was possible. The mineral impurities that had slipped into the water phase from the graphite and the bitumen-sand were removed by washing with more water. In the same way the charcoal mixture and the peat mixture were freed of the minerals by water washing. In case of peat, the water which was separated had a dark colour but even this colour could be removed when this water was again used for further washing at later stages. In the case of other mixtures the minerals coming out were rather reddish in the beginning and white towards the end. It is necessary to add that as most of the material was mineral matter and water which was washed off the kneader during the process, the remaining bitumen-graphite phase became so little that kneading was no longer efficient. Further initial portions of materials of the same ratio had to be added in order to accumulate sufficient bitumen-phase in the kneader. The pasty graphite-, charcoal-, peat-bitumen phase was then removed and the free water was removed from the paste by pressing in a filter–paper and by keeping in an open dish. A weighed quantity of this paste was then dried at 105° C. until all the moisture was driven out and then extracted with benzene to remove all the bitumen, and the bitumen-free graphite or charcoal or peat was taken for ash analyses.

The data obtained in these experiments are given in Tables I, II and III in the form of material balances, where the initial mixtures, unstable mixtures, and the final stable mixtures are shown by weight in grams and as percentage weights of each one of the systems used.

5. EXPERIMENTAL DATA

TABLE I

Material balance of Phase-exchange experiments of Bitumen-sand and Graphite

Experiment No. 1. Graphite (–325) mesh; Ash content 51·8%; Bitumen-sand: 85·0% Ash content and 15·0% Bitumen-content

	Initial Mixtures				Unstable Mixture		Stable Mixtures							
							Theoretical Separation				Actual Separation			
	Water-graphite phase		Bitumen-sand phase				Bitumen-graphite phase		Water-mineral phase		Bitumen-graphite phase		Water-mineral phase	
	g.	%	g.	%	g	%	g.	%	g.	%	g.	%	g.	%
(i) Graphite	28·2	27·8	0·0	0·0	28·2	16·7	It was found that the amount of bitumen was insufficient to form a coherent bitumen-graphite phase. So washing did not remove the minerals alone.							
Minerals	31·8	30·2	54·4	85·0	86·2	51·0								
Water ..	45·0	42·0	0·0	0·0	45·0	26·6								
Bitumen	0·0	0·0	9·6	15·0	9·6	5·7								
Total ..	105·0	100·0	64·0	100·0	169·0	100·0								
(ii) Graphite	14·1	26·8	0·0	0·0	14·1	8·1	Results same as experiment (i)							
Minerals	15·9	30·2	80·0	65·7	95·9	55·0								
Water ..	22·5	43·0	27·5	22·6	50·0	28·8								
Bitumen	0·0	0·0	14·1	11·7	14·1	8·1								
Total ..	52·5	100·0	121·6	100·0	174·1	100·0								
(iii) Graphite	15·7	18·0	0·0	0·0	15·7	5·84	15·7	36·8	0·0	0·0	15·7	30·5	0·0	0·0
Minerals	11·8	13·5	154·8	85·0	166·6	62·0	0·0	0·0	106·6	73·5	5·1	9·9	161·5	74·5
Water ..	60·0	68·5	0·0	0·0	60·0	22·3	0·0	0·0	60·0	26·5	3·7	7·2	56·3	25·5
Bitumen	0·0	0·0	27·0	15·0	27·0	10·0	27·0	63·2	0·0	0·0	27·0	52·5	0·0	0·0
Total ..	87·5	100·0	181·8	100·0	269·3	100·14	42·7	100·0	226·6	100·0	51·5	100·1	217·8	100·0

Moisture content (before extraction of bitumen) = 7·2%

Water removal = 56·3 × 100/ 60·0 = 94·0%

Ash content (after extraction of bitumen) = 24·5%

Mineral removal = 161·5 × 100/166·6 = 97·0%

TABLE II

Material balance of Phase-exchange experiments of Bitumen-sand and Charcoal

Experiment No. 2. Charcoal (−150) mesh; Ash content 5·0%; Bitumen-sand: 85·0% Ash content and 15·0% Bitumen content

<table>
<tr><th rowspan="3"></th><th colspan="4">Initial Mixtures</th><th colspan="2" rowspan="3">Unstable Mixture</th><th colspan="8">Stable Mixtures</th></tr>
<tr><th colspan="2" rowspan="2">Water-charcoal phase</th><th colspan="2" rowspan="2">Bitumen-sand phase</th><th colspan="4">Theoretical Separation</th><th colspan="4">Actual Separation</th></tr>
<tr><th colspan="2">Bitumen-charcoal phase</th><th colspan="2">Water-mineral phase</th><th colspan="2">Bitumen-charcoal phase</th><th colspan="2">Water-mineral phase</th></tr>
<tr><td></td><td>g.</td><td>%</td><td>g.</td><td>%</td><td>g.</td><td>%</td><td>g.</td><td>%</td><td>g.</td><td>%</td><td>g.</td><td>%</td><td>g.</td><td>%</td></tr>
<tr><td>(i) Charcoal</td><td>27·5</td><td>21·3</td><td>0·0</td><td>0·0</td><td>27·5</td><td>12·5</td><td colspan="8" rowspan="5">It was found that the amount of bitumen was insufficient to form a coherent paste. So washing did not remove the minerals alone.</td></tr>
<tr><td>Minerals</td><td>1·5</td><td>1·16</td><td>78·0</td><td>85·0</td><td>79·5</td><td>36·0</td></tr>
<tr><td>Water ..</td><td>100·0</td><td>77·5</td><td>0·0</td><td>0·0</td><td>100·0</td><td>45·4</td></tr>
<tr><td>Bitumen..</td><td>0·0</td><td>0·0</td><td>13·7</td><td>15·0</td><td>13·7</td><td>6·2</td></tr>
<tr><td>Total ..</td><td>129·0</td><td>99·96</td><td>91·7</td><td>100·0</td><td>220·7</td><td>100·1</td></tr>
<tr><td>(ii) Charcoal</td><td>13·75</td><td>12·0</td><td>0·0</td><td>0·0</td><td>13·75</td><td>6·65</td><td colspan="8" rowspan="5">Same as experiment (i)</td></tr>
<tr><td>Minerals</td><td>0·72</td><td>0·63</td><td>78·0</td><td>85·0</td><td>78·72</td><td>38·2</td></tr>
<tr><td>Water ..</td><td>100·0</td><td>87·5</td><td>0·0</td><td>0·0</td><td>100·0</td><td>48·5</td></tr>
<tr><td>Bitumen..</td><td>0·0</td><td>0·0</td><td>13·75</td><td>15·0</td><td>13·75</td><td>6·65</td></tr>
<tr><td>Total ..</td><td>114·47</td><td>100·13</td><td>91·75</td><td>100·0</td><td>206·22</td><td>100·0</td></tr>
<tr><td>(iii) Charcoal</td><td>14·0</td><td>18·7</td><td>0·0</td><td>0·0</td><td>14·0</td><td>5·3</td><td>14·0</td><td>33·3</td><td>0·0</td><td>0·0</td><td>14·0</td><td>10·7</td><td>0·0</td><td>0·0</td></tr>
<tr><td>Minerals</td><td>0·7</td><td>0·93</td><td>160·0</td><td>85·0</td><td>160·7</td><td>61·0</td><td>0·0</td><td>0·0</td><td>160·7</td><td>72·5</td><td>74·5</td><td>57·2</td><td>86·2</td><td>65·0</td></tr>
<tr><td>Water ..</td><td>60·0</td><td>80·5</td><td>0·0</td><td>0·0</td><td>60·0</td><td>22·8</td><td>0·0</td><td>0·0</td><td>60·0</td><td>27·5</td><td>13·8</td><td>10·6</td><td>46·2</td><td>35·0</td></tr>
<tr><td>Bitumen..</td><td>0·0</td><td>0·0</td><td>28·0</td><td>15·0</td><td>28·0</td><td>10·6</td><td>28·0</td><td>66·6</td><td>0·0</td><td>0·0</td><td>28·0</td><td>21·4</td><td>0·0</td><td>0·0</td></tr>
<tr><td>Total ..</td><td>74·7</td><td>100·13</td><td>188·0</td><td>100·0</td><td>262·7</td><td>99·7</td><td>42·0</td><td>99·9</td><td>220·7</td><td>100·0</td><td>130·3</td><td>99·9</td><td>131·8</td><td>100·0</td></tr>
<tr><td colspan="7">Moisture content (before extraction of bitumen) = 10·6%
Water removal = 46·2×100/60·0 = 77·0%</td><td colspan="8">Ash content (after extraction of bitumen) = 84.2%
Mineral removal = 86·2×100/160·7 = 53·5%</td></tr>
<tr><td>(iv) Charcoal</td><td>10·0</td><td>14·2</td><td>0·0</td><td>0·0</td><td>10·0</td><td>4·9</td><td>10·0</td><td>33·3</td><td>0·0</td><td>0·0</td><td>10·0</td><td>11·5</td><td>0·0</td><td>0·0</td></tr>
<tr><td>Minerals</td><td>0·5</td><td>0·7</td><td>113·0</td><td>85·0</td><td>113·5</td><td>55·8</td><td>0·0</td><td>0·0</td><td>113·5</td><td>61·5</td><td>50·3</td><td>58·1</td><td>63·2</td><td>53·5</td></tr>
<tr><td>Water ..</td><td>60·0</td><td>85·1</td><td>0·0</td><td>0·0</td><td>60·0</td><td>29·5</td><td>0·0</td><td>0·0</td><td>60·0</td><td>38·5</td><td>6·3</td><td>7·2</td><td>52·7</td><td>46·5</td></tr>
<tr><td>Bitumen..</td><td>0·0</td><td>0·0</td><td>20·0</td><td>15·0</td><td>20·0</td><td>9·8</td><td>20·0</td><td>66·6</td><td>0·0</td><td>0·0</td><td>20·0</td><td>23·0</td><td>0·0</td><td>0·0</td></tr>
<tr><td>Total ..</td><td>70·5</td><td>100·0</td><td>133·0</td><td>100·0</td><td>203·5</td><td>100·0</td><td>30·0</td><td>99·9</td><td>173·5</td><td>100·0</td><td>86·6</td><td>99·8</td><td>115·9</td><td>100·0</td></tr>
<tr><td colspan="7">Moisture content (before extraction of bitumen) = 7·2%
Water removal = 52·7×100/60·0 = 88·0%</td><td colspan="8">Ash content (after extraction of bitumen) = 83·4%
Mineral removal = 63·2×100/113·5 = 55·5%</td></tr>
</table>

TABLE III

Material balance of Phase-exchange experiments of Bitumen-sand and Peat

Experiment No. 8. Raw Peat: Dry peat content 21·0%; Bitumen-sand: 85·0% Ash content and 15·0% Bitumen content

	Initial Mixtures				Unstable Mixture		Stable Mixtures							
							Theoretical Separation				Actual Separation			
	Raw peat		Bitumen-sand				Bitumen-peat phase		Water-mineral phase		Bitumen-peat phase		Water-mineral phase	
	g.	%	g.	%	g.	%	g.	%	g.	%	g.	%	g.	%
Dry Peat ..	42·0	21·0	0·0	0·0	42·0	10·5	42·0	57·0	0·0	0·0	42·0	39·4	0·0	0·0
Minerals ..	0·0	0·0	168·4	84·2	168·4	42·1	0·0	0·0	168·4	51·6	9·9	9·3	158·5	54·0
Water ..	158·0	79·0	0·0	0·0	158·0	39·5	0·0	0·0	158·0	48·5	23·1	21·6	134·9	46·0
Bitumen ..	0·0	0·0	31·6	15·8	31·6	7·9	31·6	43·0	0·0	0·0	31·6	29·6	0·0	0·0
Total ..	200·0	100·0	200·0	100·0	400·0	100·0	73·6	100·0	326·4	100·0	106·6	99·9	293·4	100·0

Moisture content (before extraction of bitumen) = 21·6%

Water removal = 135·9 × 100/158·0 = 85·5%

Ash content (after extraction of bitumen) = 19·1%

Mineral removal = 158·5 × 100/168·4 = 94·3%

6. OBSERVATION AND DISCUSSION

In Experiments 1 (i), (ii) and 2 (i), (ii) it was found that the amount of bitumen that was added was not sufficient to form a continuous phase with graphite or charcoal. But it was clearly seen that the process of phase-exchange was taking place. Since no continuous bitumen-phase was formed in these above experiments a clear-cut separation of the two phases by washing was not possible. But some released mineral impurities were washed in the beginning and it was seen that the released minerals did no longer contain any bitumen.

Thus, in Experiments 1 (iii), and 2 (iii) and (iv) the quantity of bitumen was increased by the addition of more of bitumen-sand up to nearly the quantity of bitumen to graphite or charcoal. In these experiments a continuous phase was then formed, and the released minerals could be washed away easily.

In all the three cases, there is an appreciable removal of mineral material from the bitumen-phases which is highest in the case of graphite of 97%, practically identical in the case of peat of 94·3% and appreciably less in the case of charcoal of 65% only. The latter case seems to indicate that there might be some adherence between the mineral matter of bitumen-sand and the charcoal. A very remarkable finding is the high removal of water in the case of peat, of 85% of the water contained in the peat. This is the more remarkable as such water removal has been achieved by mechanical mixing only, with no evaporation. As water removal from peat by other mechanical means has always shown to be a big problem, this finding opens up a new possibility of mechanical water removal, as it is to be expected that also other suitable oils or oil residues will have a similar effect.

If the remaining ash and water contents in the oil phases are related to the pure carbonaceous solids then the following ash and water contents are found:

		Ash	Moisture
Graphite	..	24·5%	19·1%
Charcoal	(iii)	84·2%	49·6%
	(iv)	83·4%	38·6%
Peat		19·1%	35·5%

The ash contents and water contents are based on graphite, charcoal and peat.

The fact that the bitumen-phase always contains quantities of water and minerals indicates that in these processes the final mixtures are the results of an equilibrium state which is approached during the operation.[5] In order to demonstrate the underlying system, a representation with the help of tetrahedric space models for these systems has been made which are shown in Figs. 1 (*a*), 1 (*b*), 2 (*a*) and 2 (*b*). From these models it becomes clear that once the separation has been started, either high recovery of bitumen from the bitumen-sand or high removal of water and minerals from the carbonaceous phases can be achieved by choosing extreme ratios of either surplus of carbonaceous material or bitumen-phase material respectively.

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The bitumen-charcoal mixture consists of a sticky solid which spreads slowly on keeping. When dried at 100° C. and cooled, it became a very tough solid. The bitumen-peat mixture was also a sticky solid, though not as sticky as the bitumen-charcoal mixture. It also spreads on keeping, and sticks to the container. The bitumen-graphite mixture is not sticky, but is a flexible plastic solid. It does not stick to the container nor spreads on keeping.

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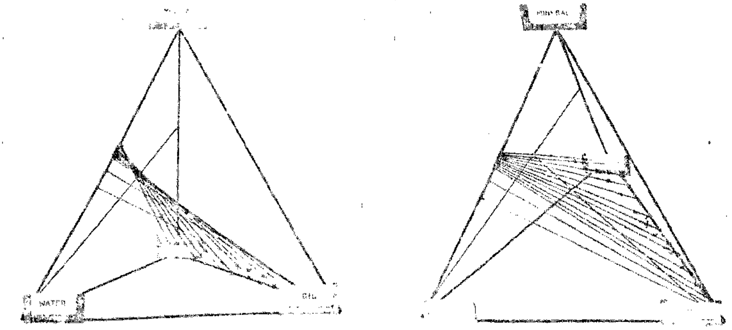

FIG. 1 (*a*) FIG. 1 (*b*)

Models of Bitumen-Sand-Graphite Mixtures

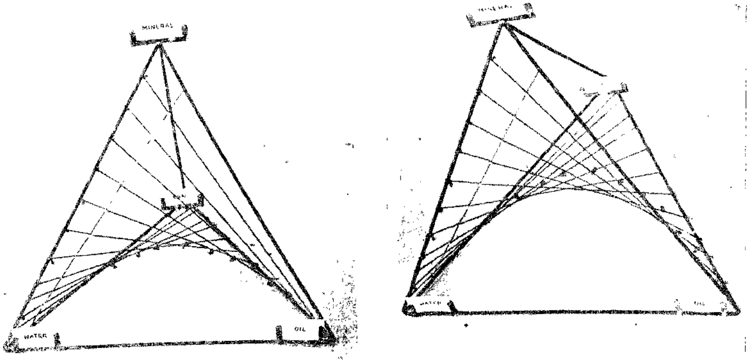

FIG. 2 (*a*) FIG. 2 (*b*)

Models of Bitumen-Sand-Peat Mixtures