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(54) Title: SOLVENT EXTRACTION AND DEWATERING OF PEAT (57) Abstract A process for dewatering and extraction of bitumen from peat comprises repeated countercurrent extraction using white spirit. A Carver-Greenfield plant may be advantageously employed.		

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SOLVENT EXTRACTION AND DEWATERING OF PEAT

This invention relates to the solvent extraction and dewatering of peat.

Peat can be dewatered and concurrently solvent extracted with organic solvents. One method of achieving this is by means of the Carver-Greenfield counter-current solvent dewatering system. This is an extremely efficient and cost-effective way of dewatering peat but is less efficient in extracting peat bitumens.

Details of the Carver-Greenfield process have been reviewed by C J Crumb and K A Crumb ('Energy from Biomas and Wastes' Symposium Orlando, Florida, 1984). US 3323575 disclosed basic details of the Carver-Greenfield process, wherein a non-volatile fluidising oil is used to form a mixture which is dehydrated by thermal evaporation. Further disclosures relating to the process are contained in US patent nos. 3539549, 3716458, 3855079, 3898134, 3917508, 3947327, 3950230, 4007094, 4013516, 4270974, reissue 31185(4276115), 4289578, 4336101, 4518458 and 4608120. GB 2060417 discloses a process wherein the oil is subsequently separated from water. A wide variety of hydrocarbon oils aliphatic alcohols and aliphatic acids are proposed. EP244233 discloses use of such a process for extraction of peat bitumens. Use of a variety of solvents is disclosed. A benzene/ethanol azeotrope was preferred to petroleum ether. Mixtures of solvents afforded higher bitumen yields than individual solvents. The effect of extraction conditions on the nature of the product was discussed in detail.

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Several solvents have been used for dewatering peat including benzene and diethyl ketone but benzene/ethanol mixtures were preferred.

The invention seeks to provide an improved method for the combined solvent extraction and dewatering of peat and to produce better yields and/or better quality peat bitumens.

According to the present invention there is provided a process for dewatering and extraction of bitumen from peat including the steps of: contacting peat with an organic solvent having an aromatic content of 6% or more and a boiling point of 120-200°C or more removing water from the peat by distillation separating the solvent with dissolved peat bitumen from residual solid matter and evaporating said solvent to yield peat bitumen.

Use of white spirit is particularly preferred. The term 'white spirit' is intended to include refined products such as is sold under the trade mark SHELL SBP11 and other petroleum hydrocarbon fractions or mixtures of boiling range 120-200°C of which the major components are alkanes and alkenes (each being either straight chain or branched chain structures), naphthenes (being polyalkyl or heterocyclic derivatives of cyclohydrocarbons whether saturated or partially unsaturated) and aromatics (including benzene, naphthalene and tricyclic aromatic compounds being alkyl, polyalkyl or heterocyclic derivatives of these ring structures). The heteroatoms in these compounds could commonly be O, N or S. The relative

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percentages of these groups of compounds can vary widely and may generally contain at least 6% aromatics. White spirit has a boiling point of 140-170°C, generally 155-168°C. The flash point is high and only a trace of benzene or sulphur compounds is normally present. Toxicity is therefore low. White spirit may commonly contain 15-25% of aromatics.

The solvent preferably forms an azeotrope with water, to facilitate removal by distillation. Preferred solvents have an aromatic content of 10°C or more, but not exceeding 40% preferably not more than 30%. The percentage of aromatics is based on the weight of components having aromatic groups.

Percentages referred to in this specification are by weight unless indicated otherwise.

The solvent is particularly suitable for use in a system such as the Carver-Greenfield counter-current solvent dewatering system. Such systems have previously employed a non-aromatic or low aromatic solvent such as low aromatic content kerosene. The use according to the invention, of a solvent having a higher aromatic content leads to a much better dewatering and extraction of peat bitumens and to production of peat bitumen extracts which are less degraded and hence have a higher value.

The top limit of the boiling range of the solvent should be no greater than about 200°C. In addition, the solvent should meet other criteria such as ease of availability, low cost, environmental and health acceptability, low sulphur content, and acceptable flashpoint and explosive characteristics.

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Selection of the solvents of this invention from the wide range of available compounds and mixtures affords a number of unique benefits and advantages which were not evident from the relevant prior art, in relation to the treatment of peat.

Volatile solvents may be considered to be generally desirable for solvent extraction on account of their easy separation from the extracted material. However the criteria for dewatering processes are different. Generally, acetone, other ketones, ethanol or other alcohols are chosen for dewatering processes. We have found that these are too polar for bitumen extraction from peat.

Selection of white spirit is beneficial. The solvating power is greater than for alkane solvents but the polarity is not as high as alcohol or ketone solvents. In addition, toxicity is low and the solvent is cheap. The physical properties are particularly suited to the Carver-Greenfield process. The boiling point of 140-170°C allows easy recovery of the solvent by distillation preferably under vacuum without pyrolysis of the residual bitumen. Moreover, countercurrent extraction requires a solvent with a higher boiling point than water but below that of the bitumen. Formation of an aqueous azeotrope having a boiling point of 90-100°C is particularly beneficial, allowing simple extractor design and processing using steam heating. Moreover, white spirit is immiscible with water facilitating separation from the latter. A further unexpected advantage is that white spirit has superior wetting properties on peat, particularly in comparison to paraffins. Slurries of peat and white

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spirit may be easily handled by pumping and similar means.

In a typical Carver-Greenfield counter-current system for the dewatering and solvent extraction of peat there will be four containers or 'pots'. Peat, typically containing 50% water, disposed within the first pot with the chosen solvent (which should of course be immiscible with water) is heated to a temperature below 100°C. A certain amount of the water will evaporate and after a fixed time the partially treated peat is moved on to a second pot where it is treated in a similar manner at a higher temperature. Four pots in all may be involved and the solvent may be introduced into pot 4 and transferred sequentially through pots 3 and 2 to pot 1 while the peat itself moves in the opposite direction. This provides efficient thermal transfer between the pots. It has been found that solvent in accordance with the invention shows markedly improved dewatering properties as well as improved bitumen extraction.

Table 1 (page 5a) gives details of two preferred solvents in accordance with the invention, Shell SBP11 and Shell White Spirit, together with two solvents previously used in the Carver-Greenfield system Esso ispoar L and Esso solvent 2012, the latter being a commercial grade of kerosene.

The solvents of the method of the invention also exhibit superior wetting rates with these being roughly in line with the aromatic content.

Two methods of extracting the peat bitumens from peat have been used: (i) Soxhlet (ii) digestion.

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In the Soxhlet extraction method the peat (about 50g) is continuously washed with clean solvent at approximately room temperature, but the syphoned solution is boiled continuously at the relatively high boiling temperature of the commercial solvent (Table 1). Hence the extracted peat bitumens are subjected to high temperatures for prolonged periods and the extract was pale yellow coloured. For the digestion procedure, about 100g of milled peat wrapped in muslin was stirred in the refluxing solvent for about three hours. The peat bitumen solution was filtered and the water layer separated from the organic solvent layer. In both cases the solvent was distilled from the peat bitumen under vacuum (0.89 torr, measured on a McLeod gauge) on a rotary evaporator, the last solvent being removed by freeze drying.

The initial and final water content of the peat was determined by the Dean and Stark method to avoid confusion between solvent and water losses if the peat residue water content is estimated by the oven method. The yields, saponification value and acid value of the peat bitumens and initial and final water contents of the peat after solvent extraction with three solvents by Soxhlet and digestion methods are given in Table 4.

In Soxhlet solvent extraction of peat, the efficiency of peat dewatering was minimal, which was in sharp contrast to peat dewatering by the digestion method for peat bitumen removal. Hence the temperature of solvent extraction is critical; compare room temperature for Soxhlet extraction and in excess of 120°C for digestion extraction. Water

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is probably vaporised from the peat in hot digestion. Also with Shell white spirit and SPB11, distinct layers developed in the filtered solvent, the lower one being water and containing acidic and other water soluble residues from the peat.

The Esso 2012 solvent was difficult to distil from the extracted peat bitumens because of its relative high upper boiling point (240°C at atmosphere pressure) when some of the peat bitumens also distil. Bumping of the solution during distillation could only be avoided by high speed agitation and probably arose from colloiddally dispersed water in the solvent. The colloidal water would explain the poor efficiency of the digestion method in removing water from peat when compared with the other solvents. The collodial water would be in equilibrium with the water saturated peat whereas in the white spirit and SBP11 very little collodial water exists; it separates into a layer or is lost as the low boiling azeotrope.

Extraction by the digestion procedure has shown that the moisture content of the peat is considerably reduced from 50%-10% (white spirit and SBP11, Table 2). However in both cases the extraction digestion was carried out at temperatures greatly exceeding the boiling point of water such that vaporisation of water occurs followed by replacement with the solvent. To investigate to what extent the peat is dewatered at lower temperatures, a peat/white spirit slurry was stirred at temperatures from 20°C to 100°C for 3 hours at each temperature. The peat was sampled at hourly intervals and the moisture content was estimated by Karl Fisher titration, (Table 3 below).

Table 3

Moisture content of peat after heating in white spirit for prolonged periods at increasing temperatures

Temperature	Moisture Content after Stirring		
	1hr	2hr	3hr
20	7.8	7.6	7.0
40	6.0	6.2	5.7
60	5.1	5.3	5.2
80	4.7	1.8	1.5
100	1.4	0.3	0.2

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After completion of the experiment the peat was filtered. It was noted that there was no water layer present in the solvent. During the experiment the liquid level in the open container dropped suggesting the water and/or solvent had evaporated. The loss was made good with white spirit.

It is evident that the moisture content decreased very slowly over the temperature range 20°C to 60°C but at 80°C there was a large drop in the moisture content and a further substantial drop at 100°C. It is believed that this was because hydrogen bonding associated with water molecules began to break down at 80°C and the rate of diffusion of both water and solvent from and into peat increased substantially as the temperature was increased. Also at the high temperature there was increased evaporation of the water from the peat/solvent mixture as the vapour pressure of the water increased which in turn disturbed the peat water solvent diffusion and hydrogen bonding equilibria.

The experiment was then modified by using a fresh peat sample at each stage and it was found that initial stirring of peat and solvent resulted in a thixotropic gel structure which moved as a mass with the stirrer and hence was very difficult to stir. (This effect was not apparent in a larger scale extraction). As the mixture lost water however, stirring became much easier due to:-

- a. chopping and cutting of the peat by the blades of the stirrer
- b. breakdown of water-peat hydrogen bonding
- c. bursting of fibres and cells as water contained therein expanded with increasing temperature.

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All peat samples were left standing in white spirit for 1 hour at room temperature and the moisture was estimated (Table 4) before agitating the slurry and raising the temperature.

At 20°C there was no appreciable water loss after 4 hours (Table 4). The yield of bitumen was low and it was a pale coloured liquid. The mixture was difficult to stir even after 3 hours maceration.

At 40°C there was no appreciable reduction in moisture level either and the mixture was difficult to stir even after 3 hours maceration. The bitumen yield increased and was less fluid.

At 60°C again no reduction in moisture level was apparent but the slurry lost its gel structure after 2 hours maceration when stirring was much easier.

At 80°C after 1 hour the stirring became easier and there was a significant reduction in the moisture level after 3 hours with further reduction after another 2 hours. The yield of bitumen did not increase significantly over the yield realised at 60°C but the solvent/peat ratio (w/w) had been reduced from 6:1 to 5:1 (it being easier to stir the slurry at the higher temperature). It is likely that the decrease in solvent offset the greater solubility of the bitumen in the solvent at increased temperatures and a relatively greater proportion of solvent containing bitumen remained on the separated peat.

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At 100°C the solvent/peat ratio (w/w) (Table 4) was further reduced to 4:1. In this case an actual reduction in yield occurred compared with the conditions at 80°C. The reduction in moisture content was very high, there being no water detectable by the Dean and Stark method. The consistency of the bitumen was indistinguishable from that recovered at 80°C.

No aqueous layer was observed for any set of operating parameters at which the experiments were carried out, either in the peat/solvent mixture or in the recovered solvent following filtration. Both the reduction in moisture level and decrease in viscosity of the peat slurry after 3 hours maceration at 80°C suggested that these were the mildest conditions necessary for the breakdown of the peat-water complex and hence efficient dewatering of peat. With data from these experiments a large scale solvent extraction of 600kg of peat was undertaken.

The large scale solvent extraction involved the use of 2200kg of Shell SBP11 solvent heated at a temperature of 85-90°C for 3 hours. The reaction vessel was not vented as a fire precaution and hence water was not removed from the system as previously. 1600kg of solvent containing peat bitumen was collected, the remaining solvent being associated with the extracted peat since no water had been removed from it during this trial. Under normal conditions the water would be removed and the remaining solvent recovered (less a small percent, typically 0.5%, remaining on the extracted peat). Making allowance for this, the recovered peat bitumens extracted amounted to 9% of the dry weight of the original peat, which contained 55% water.

The solvent was distilled from the extracted peat bitumens under reduced pressure at about 100°C.

Characteristics of the extracted bitumens

Traditionally peat bitumens contain three classes of material:

1. waxes
2. asphaltenes
3. resins

Peat bitumen is an ester-type wax. The resins present in wax are relatively more soluble in non-polar solvents such as paraffins. Various procedures have been used to determine the resin content of waxes. They involve classifying as resins those wax constituents which are soluble in cold alcohol or cold ethyl acetate. Such a classification is crude and allows for considerable overlap of constituent material.

Attempting to isolate pure compounds from the crude bitumen would not prove useful at this stage since it is known that the composition of bitumen is very complex and no one component would represent more than 1% of the bitumen. However, a broad picture of the constitution of the whole bitumen and fractions can be appreciated from the change in gross characteristics on de-resinification using the solvent fractionation scheme given in Table 5. The resin content and de-resinated wax content were similar for bitumen extracted at 40°C, 60°C and 80°C. The bitumen extracted at 20°C differed from the other bitumens in the larger resin and lower wax content. On the other hand, the bitumen from the large scale extraction gives larger wax content than resin content. The solvent used in this extraction was SBP11 rather than white spirit and may extract ester waxes in preference to resins.

There was little variation in the acid number (a measure of the free fatty acid content) of the various bitumens except for the 20°C extract which had a very low acid number. The saponification number (a measure of the total amount of esters and free acid) showed greater variation with similar values for the higher temperature extractions (80°C and large-scale), a fall for the lower temperature extractions at 60°C and 40°C and a very low value for the 20°C extract.

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After deresinification all samples showed an increase in the acid number of the wax fraction and a corresponding decrease in the acid number of the resin fraction. The saponification number of the resin fractions showed a slight drop after deresinification while the wax fractions showed a large increase in saponification number even doubling their value. These trends indicate a concentration of the ester waxes in the de-resinated wax fraction as expected but it is obvious that the fractionation is a crude one with overlap of constituents. The iodine values which are a measure of the degree of unsaturation of the waxes remained relatively constant for all experiments.

In each case the deresinated wax was harder than the crude bitumen from which it was derived. This was quantified by the increased melting points. Determination of melting point by capillary tube (sometimes called slip-point) is not normal practice for these waxes but was used in this case for simplicity. Only the large scale bitumen resin fraction had a melting point above room temperature.

Although the bitumens differed from the deresinated waxes in melting point, saponification and acid numbers their infra-red spectra were similar. The frequencies observed are listed in Table 6 together with their assignments proposed by comparison with the available literature. The proposed assignments indicate the presence of normal alkyl saturated and unsaturated esters and elements with long methylene chains. It is noteworthy that the carbonyl stretching signal, was much weaker for the resin fraction.

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By way of comparison, the composition of Esso 2012 (kerosene) extracted peat bitumen was characterised as:

waxes	8.1%
asphaltenes	0.62%
resins	91.28%

The high aromatic solvent employed in the method of the present invention enable processes such as the Carver-Greenfield peat dewatering process to operate more efficiently and in particular to give maximum yields of peat bitumen and better wetting of the peat. Moreover the composition of the peat bitumen extracted, being higher in waxes, is superior so the products are therefore more valuable commercially. The dewatered peat may be used as a fuel as is conventional and the removal of the bitumens may actually improve its utility as a fuel since it burns with less smoke.

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Figure 1 illustrates use of a Carver-Greenfield process in accordance with this invention.

Raw peat is placed in a fluidising tank 2 with the solvent, white spirit. The slurry produced is pumped into a first evaporator 6. The evaporator is heated by waste water vapour through line 14 on the second evaporator stage 7. Water vapour from the first evaporator stage 6 passes through a cooling system 3 to a vacuum system 1 and vent. Solvent condensed by the cooling system 3 is separated in the separator 4 and returned to the fluidising tank 2. Dehydrated peat in a slurry with the solvent is pumped into the second evaporator stage through the line 13. The second evaporator stage is steam heated and completes the dehydration and extraction from the peat. The pressure in the second evaporator is lower, allowing further water to be removed at a lower temperature. The slurry of dry peat and bitumen containing solvent is pumped to a centrifuge 9. The solid material from the centrifuge is passed to a de-oiling apparatus 8 which produces dry de-oiled peat 15 for use as combustible fuel. The solvent from the centrifuge and the de-oiler is recycled to a recovery system 10 wherein solvent is distilled to yield bitumen and pure solvent for re-use.

Although Figure 1 shows a two stage evaporator system three, four or more stages may be employed in accordance with conventional Carver-Greenfield practice. Deoiled peat may find use as a combustible fuel, in animal feedstuffs and may yield beneficial hydrolysis products.

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Claims

1. A process for dewatering and extraction of bitumen from peat including the steps of: contacting peat with an organic solvent having an aromatic content of 6% or more and a boiling point of 120-200°C or more, removing water from the peat by distillation, separating the solvent with dissolved peat bitumen from residual solid matter and evaporating said solvent to yield peat bitumen.
2. A process as claimed in claim 1, wherein the solvent has a boiling point of 140°C - 170°C.
3. A process as claimed in claims 1 and 2, wherein the solvent forms an azeotrope with water.
4. A process as claimed in any preceding claim wherein the solvent has an aromatic content of 10% or more.
5. A process as claimed in any preceding claim, wherein the solvent is white spirit.
6. A process as claimed in any preceding claim, wherein a slurry of peat and said solvent is repeatedly contacted with a flow of the solvent.
7. A process for dewatering and extraction of bitumen from peat which comprises forming a slurry of peat with an organic solvent having an aromatic content of 6% or more and a boiling point of 120-200°C, heating said slurry in a first container to a temperature sufficient to evaporate a portion of the water present in the peat slurry, passing the peat

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slurry to a second container and heating the peat slurry to a temperature sufficient to evaporate further water from the peat slurry utilising heat derived from the evaporated water from the previous container, and thereafter separating the solvent containing peat bitumen dissolved therein from the residual solid peat and separating the peat bitumen from the solvent.

8. A process as claimed in claim 7, in which the peat slurry is passed successively through at least three containers, the slurry being heated in each container to evaporate water using heat derived from the water evaporated in the previous container.

9. A process as claimed in claim 7 or 8, in which the slurry in the second and subsequent container is heated to a temperature lower than the previous container.

10. A process as claimed in any of claims 7 to 9, in which the pressure in the second and subsequent container is less than that in the previous container.

11. A process as claimed in any of claims 7 to 10, wherein the solvent has a boiling point of 140-170°C.

12. A process as claimed in any of claims 7 to 11 wherein the solvent is white spirit.

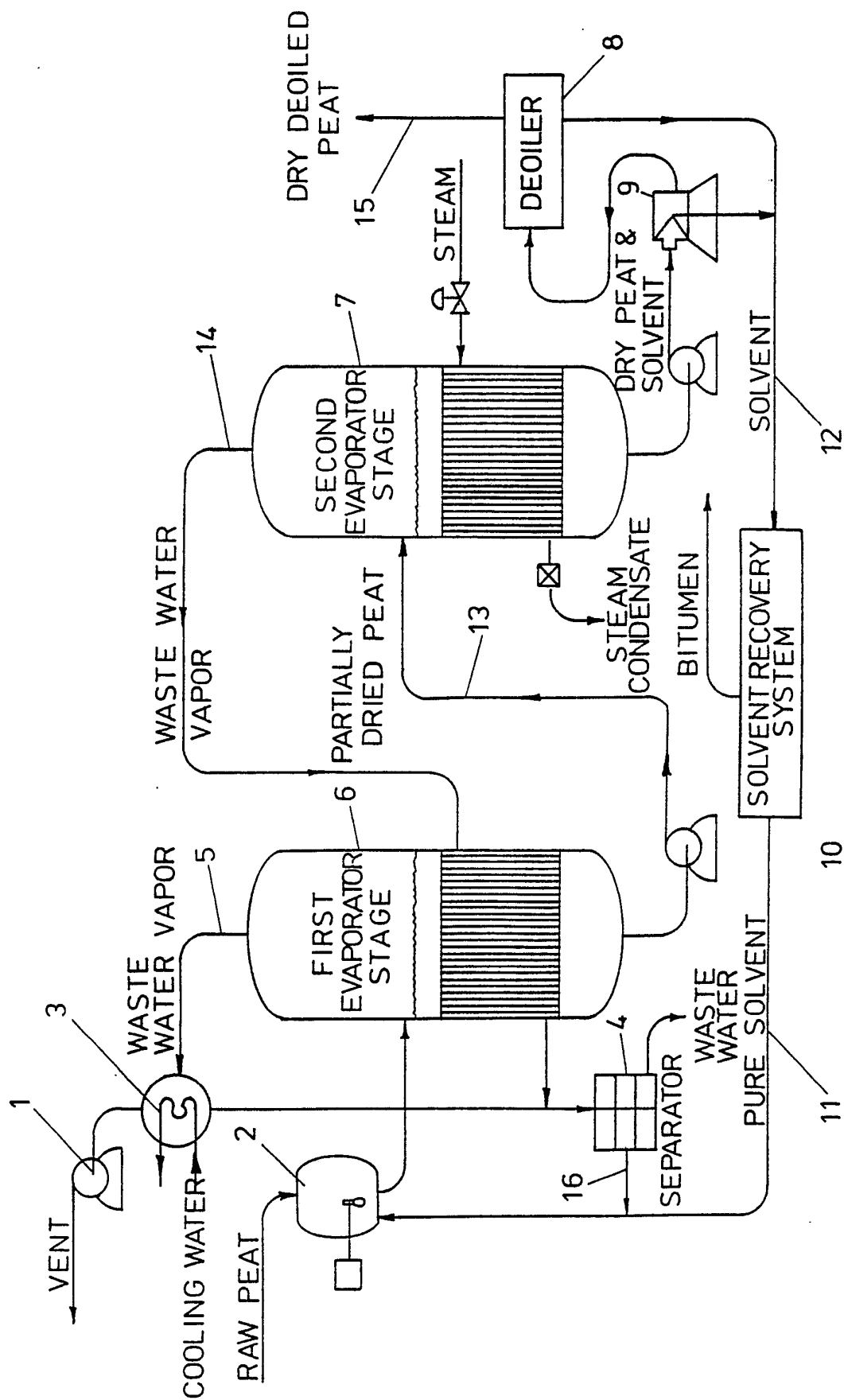


FIG. 1

TABLE 1

SOLVENT DATA

Solvent	Price £1 ⁻¹	Aroma- tics %	Flash- point °C	Boiling range °C	Explosive limits air at 28°C % vol	Density in at 15°C kg 1 ⁻¹	Sulphur Content mg kg ⁻¹
Shell SBP11	0.30	23	41	155-166	0.9-8.0	0.779	<2
Shell White Spirit	0.24	17	41	153-197	0.6-8.0	0.870	<5
Esso isopar L	0.48	0.1	63.8	188-206	0.6-6.5	0.772	1
Esso solvent 2012	0.24	7	49	155-240		0.780	300

TABLE 2

Efficiency of solvent dewatering and bitumen extraction of the solvents

Solvent (with extraction method)	% water content of peat:-		% yield of bitumen on dry peat		saponification value		acid value	
	initial	final						
Shell SBP11								
a) Soxhlet	54.3	44.5	9.98	106	24			
b) digestion	54.3	11.4	10.62	108	34			
Shell white spirit								
a) Soxhlet	53.7	46.3	9.19	99	22			
b) digestion	53.7	9.7	9.74	97	28			
Esso 2012								
a) Soxhlet	52.3	49.7	6.34	117	31			
b) digestion	52.3	31.4	7.41	110	35			

Table 4

Dewatering and bitumen extraction efficiency of white spirit at various temperatures

Temperature °C	20	40	60	80	100
Solvent/peat ratio (w/w)	6:1	6:1	6:1	5:1	4:1
Moisture Content % after					
1 hr no stirring	13.0	11.7	11.9	11.9	13.0
1 hr with stirring	12.2	12.0	11.9	10.1	9.0
2 hr with stirring	11.9	11.8	11.4	9.8	4.4
3 hr with stirring	12.9	11.8	10.3	5.3	0.9
4 hr with stirring	-	-	-	2.2	-
5 hr with stirring	-	-	-	1.5	-
Bitumen yield after 3 hrs stirring % on dry peat					
	2.6	4.34	6.0	7.0	6.3
Bitumen consistency					
	light liquid	yellow buttery	brown waxy	brown waxy	brown waxy
Final Moisture Content %					
	55	52	52	20	0

$$\frac{6}{4}$$

TABLE 5

Comparison of peat bitumen de-resinified wax and resins

	Large scale extraction				
	90°C	80°C	60°C	40°C	20°C
% Resin	35	60	53	56	83
% wax	49	32	39.6	32	2
% Residue (contain some peat fines)	12	6	6	10	12
Acid value	(34W, 12R)	(51W, 7R)	(61W, 14R)	(49W, 13R)	4
Saponification value	52	49	32	32	7
Idonine value	(21W, 21R)	(20W, 17R)	(14W, 16R)	(17W, 15E)	10
Melting point/°C	74 (86W, 42R)	58 (72W, LR)	49 (68W, LR)	47 (66W, LR)	L
(W=wax R=resin L=liquid)					

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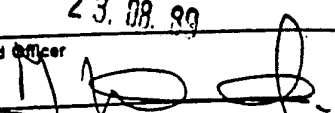
TABLE 6

ASSIGNMENT OF FREQUENCIES

Frequency cm^{-1}	Assignment
2660, 2920, 2860	Aliphatic $-\text{CH}_3$ and CH_2 stretching
1740	$-\text{CO}$ stretching (aliphatic esters)
1710	$-\text{CO}$ stretching (aromatic esters)
1610 (weak)	Aromatic ring
1470	$-\text{CH}_3$ and CH_2 plane deformation
1370	C $-\text{CH}_3$
1270	$-\text{CO}$ stretching of aromatic ethers or alcohols
725	$-\text{CH}_2$ and CH_3

INTERNATIONAL SEARCH REPORT

International Application No **PCT/GB 89/00462**

I. CLASSIFICATION OF SUBJECT MATTER (if several classification symbols apply, indicate all) ⁶		
According to International Patent Classification (IPC) or to both National Classification and IPC		
IPC ⁴ : C 10 F 5/00, C 10 G 1/04		
II. FIELDS SEARCHED		
Minimum Documentation Searched ⁷		
Classification System	Classification Symbols	
IPC ⁴	C 10 B, C 10 G	
Documentation Searched other than Minimum Documentation to the Extent that such Documents are Included in the Fields Searched ⁸		
III. DOCUMENTS CONSIDERED TO BE RELEVANT ⁹		
Category ¹⁰	Citation of Document, ¹¹ with indication, where appropriate, of the relevant passages ¹²	Relevant to Claim No. ¹³
X	FR, A, 669519 (I.G. FARBENINDUSTRIE) 18 November 1929, see abstract, points 1,2	1
Y		7-10
A	--	2-6,11,12
Y	FR, E, 36611 (I.G. FARBENINDUSTRIE) 7 July 1930, see abstract, points 1,2	7-10
A	--	1-6,11,12
A	GB, A, 844556 (C. GLINKA) 17 August 1960, see claims 1-7	1-12
A	EP, A, 0244233 (FOSTER WHEELER ENERGY) 4 November 1987, see claims 9-11 cited in the application	3-5,11,12
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	./.	
¹⁰ Special categories of cited documents: ¹⁰ "A" document defining the general state of the art which is not considered to be of particular relevance "E" earlier document but published on or after the international filing date "L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified) "O" document referring to an oral disclosure, use, exhibition or other means "P" document published prior to the international filing date but later than the priority date claimed "T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention "X" document of particular relevance: the claimed invention cannot be considered novel or cannot be considered to involve an inventive step "Y" document of particular relevance: the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art. "G" document member of the same patent family		
IV. CERTIFICATION		
Date of the Actual Completion of the International Search	Date of Mailing of this International Search Report	
23rd July 1989	23. 08. 89	
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III. DOCUMENTS CONSIDERED TO BE RELEVANT (CONTINUED FROM THE SECOND SHEET)		
Category *	Citation of Document, with indication, where appropriate, of the relevant passages	Relevant to Claim No
A	US, A, 3323575 (GREENFIELD) 6 June 1967, see claims 8-17 cited in the application -----	1,7

ANNEX TO THE INTERNATIONAL SEARCH REPORT ON INTERNATIONAL PATENT APPLICATION NO.

GB 8900462

SA 28365

This annex lists the patent family members relating to the patent documents cited in the above-mentioned international search report. The members are as contained in the European Patent Office EDP file on 10/08/89. The European Patent Office is in no way liable for these particulars which are merely given for the purpose of information.

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
FR-A- 669519		None	
FR-E- 36611		None	
GB-A- 844556		None	
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