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APPLIED SCIENTIFIC RESEARCH CORPORATION OF THAILAND

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AND ITS INDUSTRIAL APPLICATIONS

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EXTRACTION OF WAX FROM THAI LIGNITE

By Supatra Munsakul* and Bancha Udomsakdi*

SUMMARY

The contents of wax from three known lignite deposits (Li, Mae Mo, and Krabi) were studied. Laboratory Soxhlet extraction at the boiling point of various solvents were carried out. Of the solvents tried, a mixture of benzene and ethanol (50:50) was found to be the best. All crude extracts were black in colour. The effect of particle size was also investigated. The yields of crude extract were 0.9 - 4.8% (Li lignite), 0.64 - 3.00% (Mae Mo lignite), and 0.88 - 3.50% (Krabi lignite). The yields of crude wax were very low, and therefore only the crude wax from Li lignite was refined and bleached.

INTRODUCTION

No previous work on the extraction of wax from Thai lignite has been recorded. Wax from lignite has never been used directly in Thailand, but wax is known to be an ingredient in various leather and furniture polishes, imported annually into Thailand. Thailand has three known large deposits of lignite at Mae Mo, Li, and Krabi. Up to the present time only lignites at Mae Mo and Krabi have been used in electric generation while that of Li has not been fully exploited.

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MATERIALS AND METHODS

Samples of lignite were obtained from three provinces of Thailand with important lignite deposits: Lamphun (Li), Lampang (Mae Mo), and Krabi. Results of proximate analysis of the samples are shown in Table 1.

TABLE 1. PROXIMATE ANALYSIS OF LIGNITE SAMPLES

Source of sample	Proximate analysis (% by weight on air-dry basis)			
	Moisture	Volatile matter	Fixed carbon	Ash
Li	20.2	32.4	35.1	0.71
Krabi	22.5	41.9	11.6	24.05
Mae Mo	25.0	49.4	12.2	13.41

Air-dry samples of 10-mesh B.S. sieve and 72-mesh B.S. sieve were used in the extraction. Twenty grammes of the sample was taken in a Whatman extraction thimble and extractions were conducted in Soxhlet apparatus at atmospheric pressure. A variety of solvents like benzene, methanol, xylene, etc, were tried, in addition to mixed solvents: benzene-ethanol, benzene-methanol, benzene-isopropanol.

EXPERIMENTAL AND RESULTS

The solvent extraction was carried out using the standard Soxhlet apparatus consisting of a 500-ml flask, a 200-ml extraction chamber, and a condenser. The size of wax-free paper thimble used was 43 mm dia., 123 mm length. Two different sizes of lignite employed were 10 mesh B.S. sieve and 72-mesh B.S. sieve. The weight of the lignite taken in the flask chamber was about 20 g, and about 250 ml of the solvent was placed in the flask. The extraction was continued until the liquid siphoning back to the flask was colourless. After the extraction was complete, the contents of the flask were filtered to remove any lignite sample present. The filter paper was washed with the same hot solvent, the filtrate was transferred to a round-bottomed flask, and most of the solvent was distilled off. The remaining extract along with the solvent

was transferred to a previously weighed tared evaporating dish, and the residual solvent was evaporated off in an oven maintained at the boiling point of the solvent and dried to constant weight.

The data given in Tables 2-4 show the results of extracting lignite from Li, Mae Mo, and Krabi with the various solvents.

TABLE 2. SOXHLET EXTRACTION OF LIGNITE FROM LI

No.	Solvent	Size of sample (B.S. sieve)	Yield (% on air-dry lignite)	Boiling point of extractive (°C)	Time of extraction (h)	Melting point of crude wax (°C)
1.	Benzene	10	0.9	80	24	76-82
2.	Benzene	72	0.95	80	24	75-82
3.	Benzene-ethanol (50:50)	10	4.636	70	24	115-118
4.	Benzene-ethanol (50:50)	72	4.875	70	24	
5.	Benzene-ethanol (65:35)	10	3.92	66	24	171-175
6.	Benzene-ethanol (65:35)	72	4.685	66	24	
7.	Benzene-ethanol (50:20)	10	2.255	67	24	105-108
8.	Benzene-ethanol (50:20)	72	2.46	67	24	
9.	Benzene-ethanol (80:20)	10	2.415	72	24	118-122
10.	Benzene-ethanol (80:20)	72	2.625	72	24	
11.	Benzene-ethanol (2:1)	10	4.12	64	24	121-125
12.	Benzene-ethanol (2:1)	72	4.29	64	24	
13.	Benzene-ethanol (3:1)	10	3.65	67	24	110-121
14.	Benzene-ethanol (3:1)	72	3.924	67	24	
15.	Benzene-methanol (85:15)	10	1.95	80	24	
16.	Benzene-methanol (85:15)	72	2.079	80	24	
17.	Benzene-methanol (50:50)	10	3.63	58	24	
18.	Benzene-methanol (50:50)	72	3.842	58	24	

No.	Solvent	Size of sample (B.S. sieve)	Yield (% on air-dry lignite)	Boiling point of extractive (°C)	Time of extraction (h)	Melting point of crude wax (°C)
19.	Benzene-isopropanol (82:18)	10	2.70	76	24	
20.	Benzene-isopropanol (82:18)	72	2.975	76	24	
21.	Xylene	10	2.05	145	24	85-87
22.	Xylene	72	2.18	145	24	
23.	Methanol	10	0.92	65	24	
24.	Methanol	72	0.98	65	24	

TABLE 3. SOXHLET EXTRACTION OF LIGNITE FROM KRABI

No.	Solvent	Size of sample (B.S. sieve)	Yield (% on air-dry lignite)	Boiling point of extractive (°C)	Time of extraction (h)	Melting point of crude wax (°C)
1.	Benzene	10	0.88	80	24	82-85
2.	Benzene	72	1.25	80	24	85-86
3.	Benzene-ethanol (50:50)	10	2.00	67	24	128-135
4.	Benzene-ethanol (50:50)	72	2.13	67	24	
5.	Benzene-ethanol (65:35)	10	2.00	68	24	185-189
6.	Benzene-ethanol (65:35)	72	2.25	68	24	
7.	Benzene-ethanol (50:20)	10	1.5	67	24	103-106
8.	Benzene-ethanol (50:20)	72	2.0	67	24	
9.	Benzene-ethanol (80:20)	10	3.00	70	24	117-124
10.	Benzene-ethanol (80:20)	72	3.50	70	24	
11.	Benzene-methanol (50:50)	10	1.46	59	24	185-191
12.	Benzene-methanol (50:50)	72	1.50	59	24	

TABLE 4. SOXHLET EXTRACTION OF LIGNITE FROM MAE MO

No.	Solvent	Size of sample	Yield	Boiling point of extractive	Time of extraction	Melting point of crude wax
		(B.S. sieve)	(% on air-dry lignite)	(°C)	(h)	(°C)
1.	Benzene	10	0.64	80	24	82-84
2.	Benzene	72	0.84	80	24	83-86
3.	Benzene-ethanol (50:50)	10	1.189	68	24	110-115
4.	Benzene-ethanol (50:50)	72	1.453	68	24	
5.	Benzene-ethanol (65:35)	10	1.445	68	24	175-181
6.	Benzene-ethanol (65:35)	72	1.63	68	24	
7.	Benzene-ethanol (50:20)	10	1.40	68	24	99-102
8.	Benzene-ethanol (50:20)	72	1.55	68	24	
9.	Benzene-ethanol (80:20)	10	3.00	70	24	120-124
10.	Benzene-ethanol (80:20)	72	3.00	70	24	
11.	Benzene-methanol (50:50)	10	1.46	59	24	180-184
12.	Benzene-methanol (50:50)	72	1.50	59	24	

Properties of the extractives

The extractive as obtained, or the crude wax as it is often called, is a dark brown solid. It is a mixture of montan ester waxes and resins. The quantity of wax in the extractive was determined by dissolving away the resins in ether at room temperature while the wax was left out as an insoluble. The yields of extract from Li lignite varied from 0.9 to 4.87% depending on the solvents. The highest yield of 4.87% was obtained by using the 50:50 mixture of benzene and ethanol, as shown in Table 2. Other lignites from Krabi and Mae Mo gave the best yields of only 3.5% and 3.0% respectively, and the best solvent used was the 80:20 mixture of benzene and ethanol.

Purification of lignite wax

The crude waxes consisted of mixtures of ester waxes and resins. The resins are light brown to black in colour, and for certain uses they need further refining and bleaching. The crude wax of lignite from Li was extracted at atmospheric pressure using 50:50 mixture of benzene and ethanol as solvent. The yield of crude wax was 4.87%, and it was purified and bleached as described below.

One hundred grammes of the crude wax was extracted repeatedly with petroleum ether portions at room temperature until the solution was practically colourless. The residue (resins) was estimated. The petroleum ether portions were combined and evaporated to obtain a refined wax. The refined wax was bleached by chromic acid solution at 105-112°C. The solution was prepared from 100 g of chromium trioxide in 100 ml of water and 400 ml of 47% sulphuric acid. The yield of bleached wax was 0.58% of the refined wax, or 0.52% of the crude wax. The refined and bleached wax had a melting point of 73-76°C, an acid value of 73, and a saponification-value of 120.

CONCLUSIONS

Several solvents were screened and only benzene-ethanol mixtures were found to be the best and they were chosen for the extraction of Thai lignites for their contents of extractable material. The lignite samples from Li were found, by using the best solvent, to contain up to 4.87% of extractable material or crude wax. Using other solvents resulted in low yields of crude wax. The crude waxes were found to contain large percentage of resins and only a small amount of true wax. The particle size of the samples also influenced the yield of extract. The yield under identical conditions increases as the particle size of the lignite decreases.

Although the properties of wax from Thai lignites were found to be good and very similar to the imported montan wax, the yield of waxes investigated were very low. It is known in the literature that if the lignite contains less than 4% of crude wax, then it is not commercially

worthwhile to extract wax on an industrial scale. The low yields resulted in the cessation of further research investigation on its industrial application.

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