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# Evaluation of Heavy Medium Concentration from Heavy Liquid Tests for Megalopoli Low Grade

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## ABSTRACT

Lignite from Marathousa open pit in Megalopolis, Greece is characterised by it's low calorific value 199,4kJ/kg, high ash content 25.9%w/w and high moisture content 47.8% w/w. Heavy liquid separation tests of the size fraction 2-32mm have produced a lignite concentrate more than 50% by weight with ash content 13.3% w/w as it is and calorific value 306,7kJ/kg, a quality that fulfils the specifications put by the Public Power Corporation of Greece (PPC). The expected product quality from industrial application using Heavy Media Separation is calculated by mathematical modelling. The predicted separation efficiency using heavy media machines lies between 80% and 90% of the one obtained by heavy liquid tests. © 2002 SDU. All rights reserved.

Keywords: Lignite; Heavy medium separation

## 1. INTRODUCTION

Nearly 70% of the total electric power in Greece is produced by the combustion of lignite in the thermoelectric power stations (TEPS) at Ptolemaida, Kozani, Amyntaio and Megalopolis. Despite the fact that lignite is included among the non renewable energy resources and it is regarded as a poor quality solid fuel of particularly low thermal efficiency, the amount of the total potential deposits in Greece, about 11 billion tons, erases any dispute of it's economic significance (Kavouridis and Galetakis, 1996; Koukouzas, Foscolos, and Kotiv, 1997).

The present study is focused on the necessity to support the use of lignite as the primary energy resource by researching methods of increasing its thermal efficiency and minimizing the environmental pollution impact (LDK-Consultants, Engineers & Planners, 1995). Until today lignite is burned without previous enrichment resulting in low combustion efficiencies (25-30%) and moreover in causing environmental pollution problems mainly due to the fly ash (Kavouridis, Kostakis, and Foskolos, 1991; LDK-Consultants, Engineers & Planners, 1995), which in many cases escapes from the existing electrostatic filters due to ash overload conditions. The problems caused by the current quality of lignite, can be stated as followed:

- Low combustion efficiencies due to the low calorific content
- Increased feed rate of lignite required to satisfy TEPS nominal electric power
- Difficulties to maintain a stable fuel quality
- High quantities of fly ash produced
- High maintenance and operating costs of the mechanical equipment
- Deposition and scaling phenomena in the combustion chambers and piping due to the chemical composition of the residual ash
- Air pollution from the finer particles of the fly ash and the oxides of the produced combustion gases (SO<sub>2</sub>, NO<sub>x</sub>).

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In the last 15 years the effort for qualitative upgrading of lignite, using physical mineral processing methods, was focused upon gravity concentration and selective liberation by size reduction of poor quality fuel lignite deposits (Goutzamani, 1994; Kitsikopoulos and Markoulis, 1992). Any positive results that can be obtained by the above techniques promise significant benefits that will come from the increased combustion efficiency. These include the reduction of lignite mass required per kWh produced as well as the reduction of the maintenance and operating cost. The low ash content will also reduce the scaling and deposition phenomena in the combustion chamber and piping (Delligiannis, 1992).

The present work deals with the upgrading of Megalopolis lignite by gravity concentration. Heavy liquid analysis and prediction of the separation efficiency of a heavy media separation process formed the basis on which the course of the study was designed. The criteria set for the evaluation of the test results are related to:

- the content of ash and detrimental components in the products,
- the weight recovery of clean lignite,
- the distribution of the ash in the concentrate and the tailings,
- the recovery of the combustible matter,
- the comparison between heavy liquid test results and estimated Heavy Media plant performance.

These characteristics combined with the specified properties of the lignite to be burned in conventional steam generating electric power stations will form the final conclusions.

# 2. EXPERIMENTAL

The samples used for the test work come from the Marathousa open pit in Megalopolis, Greece operated by the Public Power Corporation.

After impact crushing and sieve sizing the appropriate size fractions were treated in heavy organic liquid baths to separate light from heavy constituents of the ore. The heavy organic liquids were mixtures of tetrabromoethane, carbon tetrachloride and propanol at appropriate proportions in order to obtain densities from 1.2g/cm<sup>3</sup> up to 2.1g/cm<sup>3</sup>. Feed fractions and separation products were assayed for moisture, different elements, ash and calorific value.

X-ray fluorescence (XRF) was used for the analyses of metal elements. For proximate, ultimate and calorific values the samples were ground to minus 150µm and analyzed following the procedures outlined by ASTM (1978). Sulphur was determined by a LECO apparatus as outlined by Foscolos and Barefoot (1970).

Ash samples were produced at 350°C, 780°C and 1050°C. The mineralogical analyses of the ash and the inorganic matter in lignite were performed by X-ray diffraction (XRD). For lignite the sample was treated with Hydrogen Peroxide ( $H_2O_2$ ) prior to XRD in order to diminish the organic matter.

## 3. DESCRIPTION OF THE LIGNITE SAMPLE

## 3.1. Proximate and elemental analyses

The contents of moisture, ash and combustible matter of the r.o.m. sample are presented in Table 1. These values have been calculated from the sub-fractions of table 4 and practically are the same with the average measured ones. The elemental analyses of the r.o.m. sample are presented in Table 2.

The high contents of ash 25.9% (w/w) and moisture 47.8% (w/w) reduce the combustible matter content to only 26.3% (w/w). Fuel is considered to be all the combustible matter of lignite (organic volatile components and fixed carbon). The ash content is particularly high as the acceptable upper limit is only 18% (w/w).

The content in organic carbon, 11.3% (w/w) is very low and the net calorific value (NCV=199.4 kJ/kg) is under the value 240kJ/kg of the average lignite mined in western Macedonia in Greece (Kavouridis *et al.*, 1991).

The contents of nitrogen and hydrogen are typical for Greek lignite (Papanicolaou, 2001). The high percentage of total sulphur, 1.4% (w/w) also affects the quality of the fuel.

Table 1									
Moisture, ash and combustible matter analysis of R.O.M. lignite sample									
Moisture Content, %	Ash Content, %	(Fixed Carbon + Volatiles), %							
47.8	25.9	26.3							

Table 2

Elemental analysis of R.O.M. lignite sample

C <sub>tot.</sub>	C <sub>org.</sub> %	*H <sub>tot.</sub> %	O <sub>tot.</sub> %	N <sub>org.</sub> %	S <sub>tot.</sub>	GCV kJ/kg	NCV kJ/kg
12.83	11,31	1.38	6.08	0.53	1.39	283.3	199.9

\* moisture not included

## 3.2. Mineralogical analysis

The minerals determined in raw lignite are quartz, albite, gypsum, muscovite, anhydrite and chlorites. The existence of hematite, illite and calcite was not clearly certified here. The same minerals were found in the ashes produced at 350°C and 780°C except for gypsum, which was dehydrated to form anhydrite. Minerals such as hematite, illite and calcite were more distinct in these X-ray diffraction diagrams.

## 3.3. Chemical analysis of the 1050°C ash

The results are presented in Table 3. All values are calculated on a dry ash basis. The dominant oxides are SiO<sub>2</sub> (53.9%, w/w), Al<sub>2</sub>O<sub>3</sub> (15.8%, w/w), Fe<sub>2</sub>O<sub>3</sub> (11.0%, w/w) and SO<sub>3</sub> (7.1%, w/w). The last one indicates the high anhydrite content of the ash. The contents of NaO, P<sub>2</sub>O<sub>5</sub>, and MnO are in low levels.

The tendency of the ash for scaling and deposition, on the combustion chamber and piping surfaces, can be evaluated by the weight ratio of the basic to acidic oxides (B/A) (Delligiannis, 1992)

$$(Fe_2O_3+CaO+MgO+Na_2O+K_2O) / (SiO_2+Al_2O_3+TiO_2)$$
(1)

This ratio should be in the range 0.1-0.3. The value obtained is 0.306, which is fairly close to the upper acceptable limit.

Table 3 1050°C ash chemical analysis (%)

- 2				010 (70)								
	Fe <sub>2</sub> O <sub>3</sub>	SiO <sub>2</sub>	$Al_2O_3$	MgO	CaO	Na₂O	K <sub>2</sub> O	TiO <sub>2</sub>	MnO	$P_2O_5$	SO₃	lgn. Loss
	11.02	53.91	15.77	3.93	4.96	0.63	1.15	1.06	0.35	0.12	7.08	3.23

## 3.4. Particle size analysis

The particle size analysis of the r.o.m. lignite sample after crushing is presented at Table 4. The moisture content increases in the coarser particles while the ash content increases in the finer particles. Carbon and sulphur also increase in coarse sizes. There is no clear distribution tendency for the other properties.

Size analysis	s of the K.C	J.IVI. Sam	Sie (alter C	rusning i	0 <i>-</i> 36mm)						
Fraction	Mean	Weight	Moisture	Ash	Combustible	C <sub>org.</sub>	$\mathbf{S}_{\text{tot.}}$	N <sub>org.</sub>	$H_{\text{tot.}}$	GCV	NCV
mm	Size	%	Content	Content	Matter	%	%	%	%	kJ/kg	kJ/kg
	mm		%	%	%						
32											
16	24	49.86	49.69	23.72	26.59	13.29	1.43	0.58	1.37	282.1	195.8
8	12	26.01	47.95	26.01	26.04	13.13	1.43	0.58	1.40	285.4	201.8
4	6	8.39	47.11	27.36	25.53	11.98	1.38	0.56	1.39	280.4	198.5
2	3	4.85	45.37	27.81	26.82	12.19	1.31	0.54	1.41	287.3	207.8
1	1.50	3.91	44.78	29.89	25.33	13.84	1.20	0.52	1.38	296.4	218.1
0.50	0.75	2.93	43.12	29.55	27.33	12.74	1.17	0.47	1.43	262.2	184.9
0.25	0.38	2.21	37.31	38.50	24.19	13.69	1.22	0.50	1.49	287.1	219.7
-0.25	0.19	1.84	33.31	41.12	25.57	14.24	0.97	0.49	1.55	268.9	206.4
TOTAL Ca	alculated	100.0	47.85	25.88	26.27	13.12	1.39	0.57	1.39	282.8	199.4

Size analysis of the R.O.M. sample (after crushing to -38mm)

## 4. HEAVY LIQUIDS SEPARATION

Table 4

#### 4.1. Separation process and results

The fractions 8-32mm, 2-8mm and 0.5-2mm, were subjected to heavy liquid separation at densities  $1.2g/cm^3$  to  $2.1g/cm^3$  and step  $0.1g/cm^3$  depending on the size fraction.

As expected the results show that lignite is concentrated to the light density fractions while the inorganic matter to the heavier ones. As the density increases the weight of the lignite product increases but its quality deteriorates. Assuming an acceptable ash content less than 15% (w/w) in the product (floats) the separation density is 1.35g/cm<sup>3</sup> for the fractions 2-8mm and 8-32mm and 1.8g/cm<sup>3</sup> for the fraction 0.5-2mm. The cumulative float and sink products at these densities are presented in Table 5.

In an industrial application of heavy media separation the overall fraction 2-32mm is of practical importance. As seen from Table 4 this fraction makes about 89.1% (w/w) of the total r.o.m ore and is efficiently separated by heavy media cyclones for example. The finer fraction - 2mm in size is less profitable to treat (Fragiskos, 1990; Fragiskos and Katrakis, 1979; Stamboliadis, 1991).

Using the results of Table 5 one can calculate Tables 6 and 7 for the fraction 2-32mm and draw some conclusions used in the discussion.

Fraction	Mean Size	Gravimetric	Weight <i>l</i>	Moisture Content	Ash Content	Combu- stible Matter	C <sub>org.</sub>	S <sub>tot.</sub>	N <sub>org.</sub>	H <sub>tot.</sub>	GCV k]/kg	NCV k]/kg
mm	mm	Fractions	%	%	%	%	%	%	%	%	<i>/</i> -	<i>,</i> - <b>-</b>
		Heavy 1.35	43.00	35.74	47.01	17.25	8.49	0.87	0.40	0.99	142.3	39.2
8 - 32	20	Light 1.35	57.00	55.82	13.01	31.17	16.82	1.85	0.71	1.67	389.3	317.6
		Total	100.00	47.19	27.63	25.18	13.24	1.43	0.58	1.38	283.0	198.0
		Heavy 1.35	47.18	38.82	41.87	19.31	6.69	0.85	0.42	1.08	165.3	90.5
2 - 8	5	Light 1.35	52.82	51.09	14.76	34.15	16.85	1.80	0.67	1.68	388.1	238.8
		Total	100.00	45.30	27.55	27.15	12.06	1.35	0.55	1.40	283.0	201.8
		Heavy 1.8	49.05	33.77	46.33	19.90	9.65	0.91	0.43	1.26	171.2	92.0
0.5 - 2	1.25	Light 1.8	50.95	47.18	15.12	37.70	16.95	1.45	0.57	1.54	388.3	316.0
		Total	100.00	40.60	30.43	28.97	13.37	1.18	0.50	1.40	281.8	203.7

Table 5 Presentation of heavy liquid analysis results

#### Table 6

Heavy liquid test results (fraction 2-32mm)

	Fractional	Initial	Moisture	Ash	Combu-						
Fraction	Weight	Weight	Content	Content	stible	C <sub>org.</sub>	S <sub>tot.</sub>	N <sub>org.</sub>	H <sub>tot.</sub>	GCV	NCV
	%	%	%	%	Matter %	%	%	%	%	kJ/kg	kJ/kg
Feed	100.00	89.11	46.91	27.62	25.48	13.06	1.42	0.57	1.38	283.1	193.6
Conc (floats)	53.72	50.24	55.16	13.25	31.58	16.82	1.84	0.70	1.67	389.1	306.7
Tail (sinks)	46.28	38.87	39.96	46.18	17.58	8.2	0.87	0.40	1.00	146.0	47.4

Table 7

Heavy liquid test element distribution, % (fraction 2-32mm)

Fraction	Weight	Moisture	Ash	Combu- stible Matter	C <sub>org.</sub>	S <sub>tot.</sub>	N <sub>org.</sub>	H <sub>tot.</sub>	GCV	NCV
Feed	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00
Conc. (floats)	53.72	66.3	27.1	69.9	72.6	73.3	69.3	68.3	77.5	89.3
Tail (sinks)	46.28	33.7	72.9	30.1	27.4	26.7	30.7	31.7	22.5	10.7

# 4.2. Discussion of the heavy liquid separation results

For the sake of a brief and comprehensive presentation, the results of the heavy liquid separation are summarised into cumulative percentages of the lighter and heavier material at the examined separation density for each size correspondingly. Table 6 shows that a feed 100% by weight with 27.6% (w/w) initial ash content, 46.9% (w/w) moisture and 25.5% (w/w fuel matter, can produce a lignite concentrate 53.4% by weight with 13.3% (w/w) ash, 55.2% w/w moisture and 31.6% (w/w) fuel matter. 'Feed' refers to the 2-32mm lignite fraction, 'Concentrate' stands for the floating fraction of the feed and 'Tailings' stands for the sinking fraction at the density of separation which is  $1.35g/cm^3$  as mentioned above.

According to Table 7, ash is distributed by 27.1% to the concentrate (floats), while the rest 72.9% is rejected. On the contrary moisture is preferentially distributed to the lignite concentrate by 66.3%. This is expected since the organic matter contains more moisture than the inorganic matter that is heavier and goes to the tailings (sinks). The fuel matter is preferentially distributed to the light concentrate by 69.92%.

A weak point for the efficiency of the separation process is the high content of total sulphur 1.84% w/w in the concentrate. This indicates that most of the sulphur is organically bonded in the lignite mass.

In general the components related to the organic matter such as fuel content, organic carbon, sulphur, nitrogen, and hydrogen respond to the concentrate. The NCV from 193.6kJ/kg in the 2-32mm fraction is considerably increased to 306.7kJ/kg in the concentrate with 89.3% recovery.

Briefly, after heavy liquid separation, the concentrate has twice as much combustible matter, almost double calorific value and half ash content compared to the initial feed. This ensures higher combustion efficiency, lower production cost per kWh and less environmental impact.

## 4.3. Chemical analysis of the 1050°C ash

Chemical assays of the 1050°C ash were performed only for the separation products of the fraction 2-8mm and are presented in Table 7. Some differences are observed between the ash of the concentrate and that of the tail. From Table 8 it is clear that the concentrate ash contains more Fe<sub>2</sub>O<sub>3</sub>, CaO and SO<sub>3</sub> than the tailings ash while there aren't any significant differences for MgO and Al<sub>2</sub>O<sub>3</sub>. The concentration of SiO<sub>2</sub> in the tailings ash is higher than in the concentrate ash.

The value of the basic to acidic oxides in the concentrate ash, as defined above, is calculated to be 0.34. It fairly exceeds the acceptable maximum and shows a low tendency for scaling and deposition (Delligiannis, 1992).

Table 8 Concentrate and tailing ash chemical analysis (fraction 2 - 8mm)  $P_2O_5$ Na<sub>2</sub>O SO<sub>3</sub> Fe<sub>2</sub>O<sub>3</sub> MnO TiO<sub>2</sub> CaO K<sub>2</sub>O SiO<sub>2</sub>  $Al_2O_3$ MgO lgnit. Fraction B/A % % % % % % % % % % % Loss, % Conc. 0.04 0.98 6.47 1.64 50.02 17.00 11.14 3.97 0.28 0.13 8.32 3.61 0.34 (floats) Tail 7.93 0.04 1.07 2.86 2.12 60.98 17.12 3.54 0.21 0.43 3.68 2.04 0.21 (sinks)

#### 5. PREDICTION OF THE HEAVY MEDIUM SEPARATION

#### 5.1 Theoretical background

The above results are obtained by heavy liquid tests under ideal separation conditions. This means that all particles of density d heavier than the liquid density d' respond to the sinks by 100% (partition coefficient H(d)=100) and correspondingly all lighter particles respond to the sinks by 0% (H(d)=0). In this case the partition curve that gives H(d) as a function of d is a horizontal line at H(d)=0 for d<d', it becomes a vertical line at d=d' and returns to horizontal at H(d)=100 for d>d' (Fragiskos, 1990).

In industrial application, for the size fraction 2-32mm, the most probable machine to be used for the sink float separation is a heavy media cyclone. The partition curve (or Tromp curve) of such equipment has to be taken into consideration to estimate the quality of the expected products.

The quantity  $Ep=(d_{75}-d_{25})/2$  of the expected partition curve is used as an index of the separation efficiency. Acceptable values of Ep are between 0.04 and 0.08 (Fragiskos, 1990; Loveday, 1970; Wills, 1993). B.K. Loveday (1970) has developed a mathematical model for the calculation of the expected partition curve that is used here to calculate the quality of the expected lignite concentrate and tailing from a heavy media plant with Ep=0.07.

According to this model:

Table O

H(d)=50exp[(d-d')/z]	for d<ď	(2a)
H(d)=50	for d=d'	(2b)
$H(d) = 100-50 \exp[(d-d')/z]$	for d>d'	(2c)
z = -Ep/ln0.5.		(3)

After that, Tables 9 and 10 are constructed to give the expected quality of the lignite concentrate (Float) and tailing (Sink) for three different densities of heavy medium (1.25, 1.30, and  $1.35g/cm^3$ ) and two size fractions (2-8mm and 8-32mm).

Table 9			
Heavy medium	separation	efficiencv	(floats)

	Separation		Moisture	Ash	Combustible	Ash	Ash(as it is)	Organic
Fraction	Density	Weight	(as it is)	(as it is)	Matter	Content	Distribution	Efficiency
mm	g/cm³	%	%	%	(as it is)	(dry basis)	%	%
					%	%		
	1.25	49.20	55.46	12.85	31.69	28.85	22.88	89.45
8 - 32	1.30	54.74	54.45	14.54	31.01	31.92	28.81	91.23
	1.35	61.12	53.20	17.92	28.88	38.29	28.46	88.57
	1.25	36.13	51.52	14.71	33.97	30.34	19.29	66.91
2 - 8	1.30	45.10	50.54	16.17	33.29	32.69	26.47	78.70
	1.35	53.76	49.68	16.72	33.60	33.60	27.29	86.70

Sinking part	icles neavy m	iedia separatio	on efficiency				
	Separation		Moisture	Ash Content	Combustible	Ash Content	Ash Content
Fraction	Density	Weight	Content	(as it is)	Matter	(dry basis)	Distribution
mm	g/cm <sup>3</sup>	%	%	%	%	%	(as it is), %
	1.25	50.80	38.54	41.94	19.52	68.24	77.11
8 - 32	1.30	45.26	37.69	43.46	18.85	69.70	71.19
	1.35	38.88	23.49	44.77	18.32	70.97	71.54
	1.25	63.87	41.78	34.82	23.40	59.81	80.72
2 - 8	1.30	54.90	41.01	36.91	22.08	62.57	73.54
	1.35	46.24	34.58	38.76	21.03	64.83	72.71



Table 10 Sinking particles' heavy media separation efficiency

Figure 1. Partition curves for Ep=0.07 and separation densities 1.25, 1.30 and 1.35g/cm<sup>3</sup>

# 5.2. Discussion of the heavy medium prediction

The partition curves corresponding to the three separation densities are presented in Figure 1. From Tables 9 and 10 one can observe that the results do not differ substantially from those obtained in the initial tests. The predicted separation efficiency (organic efficiency) using heavy media machines lies between 80% and 90%. The organic efficiency is defined as the percent ratio of the weight of the lignite produced by Heavy Media plant to the theoretical weight obtained by Heavy Liquid tests for the same ash content. The basic conclusions are the same as for heavy liquids with some quantitative adjustments that do not change the picture and are useful for more accurate predictions. It is obvious that as the separation density increases more concentrate is obtained but the quality deteriorates. Density 1.3g/cm<sup>3</sup> is considered to be the optimum case for all the material in the size range 2-32mm.

# 6. CONCLUSIONS

The conclusions of the above study can be summarized to the following:

• Heavy media separation can be used for the beneficiation of low quality lignite from Megalopolis.

- The expected quality of the product is: moisture ~50%, ash ~15% and NCV ~285kJ/kg, which fulfils the specifications (NCV  $\geq$ 240kJ/kg).
- The expected benefits are: lower production cost per kWh, higher combustion efficiency and lower environmental impact.

## 7. RECOMMENDATIONS

- The proposed unit for the beneficiation of low quality lignite from Megalopolis is a Heavy Medium cyclone treating the fraction 2-32mm at a density about 1.3g/cm<sup>3</sup>.
- For the problem of increased sulphur in the concentrate the use of CaO could be proposed in the first instance but some research for the nature of the S could produce a better solution.
- Considering that in larger sizes the cost of processing is reduced and the potential efficiency of the plant is increased, it is worth to examine the liberation at larger particle sizes.
- Even though the percentage of the fraction 0.5-2mm is relatively small, one could probably try to recover lignite by some other gravity separation process or by froth flotation.

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