

OIL AGGLOMERATION  
BENEFICIATION OF  
ZONGULDAK COAL FINES

A MASTER'S THESIS  
In  
Mining Engineering  
Middle East Technical University

By

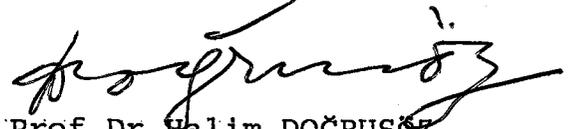
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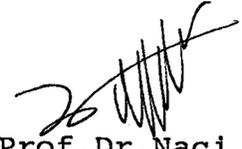
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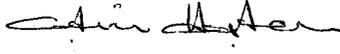
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ABSTRACT

OIL AGGLOMERATION  
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In recent years, increased attention is being paid to the application of oil agglomeration technique for improved recovery of low ash clean coal and effective beneficiation of low-grade coking coals.

In this work an experimental evaluation of the oil agglomeration process is described. Results are presented of a study in agglomeration of two kinds of coal which were flotation feed sample and jig-middlings sample from Çatalağzı coal washery. The effects of critical variables such as dosage of oil, pulp density, feed fineness and type of oil on the performance of the process were studied in detail both on flotation feed sample and middlings sample. A series of conventional flotation tests were also conducted to make a comparison between the oil agglomeration and flotation process. Flotation experiments were carried out on the flotation feed sample only.

The results suggested that dosage of oil controls the ash content of the oil agglomerated coal product; ash content of agglomerates is not sensitive to variations in pulp density; fine grinding is necessary in order to obtain lower ash agglomerates; the type of oil has no significant influence on oil agglomeration efficiency and oil agglomeration is a more selective process than froth flotation.



Key words : Oil agglomeration, flotation feed coal, jig-middlings coal, flotation.

## ÖZET

### ZONGULDAK İNCE KÖMÜRLERİNİN YAĞ AGGLOMERASYONUyla DEĞERLENDİRİLMESİ

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Son yıllarda aglomerasyon yöntemi düşük küllü kömürün yüksek verimle elde edilmesine ve yüksek küllü koklaşabilir kömürlerin değerlendirilmesinde uygulanmaya çalışılmaktadır.

Bu çalışmada Çatalağzı lavuarından alınan flotasyona giren brüt kömür ve jig çıkışı ara ürün numunelerinin aglomerasyonu işlemi ve sonuçları sunulmuştur. Her iki kömür numunesi üzerinde yağ miktarının, pülp yoğunluğunun, tane boyutunun ve yağ tipinin aglomerasyonun performansı üzerine etkisi ayrıntılı olarak incelendi. Aglomerasyon ve flotasyon işlemleri arasında bir karşılaştırma yapabilmek için bir grup flotasyon deneyleri yapıldı. Flotasyon deneylerinde sadece flotasyona giren brüt kömür numunesi kullanıldı.

Sonuçlar kullanılan yağ miktarının aglomeratların kül miktarını belirlediğini, aglomeratların kül miktarının pülp yoğunluğundaki değişmelere karşın sabit kaldığı, ince tane boyutu ile düşük küllü aglomeratların elde edilebildiğini, yağ tipinin aglomerasyonda etkisinin önemsiz olduğunu ve aglomerasyonun flotasyona göre daha düşük küllü kömürler üretebildiğini göstermiştir.

Anahtar kelimeler : Yağ aglomerasyonu, flotasyona giren brüt kömür, jig ara ürünü kömür, flotasyon.

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## 1. INTRODUCTION

The oil agglomeration process provides a viable method for recovering and up-grading fine solids from suspensions. The technique is based on the fact that finely divided solids in liquid suspension may be agglomerated and separated from the suspension by agitating a small amount of a second liquid with the mixture. The second liquid must be substantially immiscible with the suspension and preferentially wet some of the particles in the suspension.

Most coal cleaning methods depend upon the density difference between coal and its impurities to affect separation. These gravity methods, however, are not practical for particles finer than about 100 mesh (150 micron) and cleaning methods depending upon the differences in the surface chemistry of coal and foreign matter are used for the finer sizes. Froth flotation is currently the most important technique for cleaning and recovering the fine sizes of coals. However, the flotation process becomes less effective where extremely fine sizes of coal exist or if there is considerable clay slime present. The oil agglomeration process provides an attractive method for the cleaning and recovery of fine coals in the form of compact, oil-bonded aggregates.

A number of reasons may be cited why oil agglomeration is particularly well-suited to coal preparation. Firstly, coal particles are generally hydrophobic in nature and there is therefore no need for special surface conditioning agents. Secondly, the carbonaceous constituents in coal can readily be agglomerated with many different hydrocarbons. Thirdly, the agglomeration technique is very efficient in handling extremely fine sizes. Thus fines produced by natural degradation, increasingly-mechanised mining techniques, or by the grinding necessary to liberate finely-disseminated impurities from the lower quality coals being processed today, may be reconstituted into a high quality pelletized product of consistent properties.

In Turkey, coals having coking characteristics are found only in Zonguldak Coal District. These coals have been depleting fast owing to their limited reserves. So, these coals have to be cleaned more selectively and efficiently. The object of the present work was to test the suitability of the oil agglomeration process to Zonguldak coals and to compare its effectiveness with conventional froth flotation method in terms of recovery of combustibles and ash rejection.

## 2. COAL CLEANING

Coal cleaning is a generic term that is used to designate various operations performed on run-of-mine (ROM) coal to prepare it for specific end uses, such as feed to a coke oven or a coal-fired boiler or use in a coal conversion process. It is also referred to as coal preparation or coal beneficiation. Over the years, coal cleaning has come to encompass the entire spectrum of operations ranging from the relatively simple crushing and size classification operations to rather elaborate chemical and microbiological processes that are used (or are being developed) to render the ROM coal more suitable for the end use.

Coal cleaning offers a significant means of reducing the sulfur and mineral matter content of several coals thereby offering a plausible and, in many cases, economic solution to the problem of burning high-sulfur coals in an environmentally acceptable manner. Moreover, cleaning the coal also result in:

- lower transportation costs per unit of energy delivered;
- lower waste disposal costs at the user facility;
- improved coal handling capabilities;
- higher combustion efficiency in the furnace; and
- reduced slagging in the furnace, which translates into reduced downtime for the furnace.

## 2.1. The Inert Matter in Coal

When coal is ignited, it does not burn completely away. There is always some material in the coal which is inert to combustion. This material consists of water (moisture) and mineral matter which remains in an altered form as ash on the grate. The object of coal preparation is to reduce the amount of this inert material to a value acceptable to the consumer at the least cost to the producer.

Both moisture and mineral matter can be subdivided into inherent and extraneous types.

### 2.1.1. Moisture

Fresh coal at any dry place in a mine normally is saturated with moisture even though it appears dry and dusty when crushed. The percentage of moisture present, commonly called bed moisture, is more or less constant throughout a given mine and is a characteristic of rank. Such moisture varies from 1, 2, or 3% in bituminous coal to 45% or more in lignite.

Moisture is present in coal on the surface and inside cracks and large capillaries (free or surface water) or in the internal pore structure of the coal (bound or inherent water). Free water has a normal vapor pressure and the same physical properties as ordinary water. The physically held water in the internal pore structure of the coals has a vapor pressure lower than normal.

Moisture must be transported, handled, and stored; its presence in large amounts increases cost and difficulty of these operations. Looked at in another way, moisture replaces an equal amount of combustible material and thus decreases the heating value. In addition, some heat liberated in the furnace evaporates moisture in the fuel and superheats the vapor (McClung and Geer, 1979).

#### 2.1.2. Mineral matter

Mineral matter is the inert solid material in coal, and like moisture it reduces the heating value of coal by dilution. On burning coal, the mineral matter remains behind in a slightly altered form as ash. Mineral matter can be found in various forms as described in the following:

##### Inherent mineral matter :

Inherent mineral matter is the mineral matter intimately mixed with the coal. It consists of the minerals present in the original vegetation from which the coal was formed, and finely divided clays and similar materials carried into swamp by water or by wind. These clays are intimately mixed with the coal substance, and cannot be removed by coal preparation techniques.

##### Extraneous mineral matter :

Extraneous mineral matter consists of dirt bands and lenses in the seam, and shales, sandstones and intermediate rocks introduced into the mined product from

the roof and floor of the seam. Most of this material is free, and easily removed by coal preparation techniques. In some cases the dirt is strongly attached to the coal, but can be largely freed from the coal by finer crushing.

Other types of mineral matter :

Other forms of extraneous mineral matter are pyrites, and ankerite or calcite (thin white flakes often found in the joints of coals, and sometimes in the bedding plane). These are secondary minerals, deposited in the coal seam after its formation. Both of these mineral types can be so finely disseminated in the coal substance, as to be considered a part of the inherent mineral matter from a coal preparation aspect (Horsfall, 1980).

## 2.2. Coal Cleaning Processes

In general, coal preparation yields a more uniform quality fuel of greater heating value (Btu's) by reducing ash and trace elements (in some instances), removing mining wastes, and concentrating fixed carbon.

Today, coal preparation plants incorporate a variety of systems, ranging from those designed simply to remove coarse refuse from raw coal to sophisticated systems designed to remove the maximum amount of pyritic sulfur and ash. Thus a coal cleaning plant does not employ a specific process, but rather a number of different operations applied sequentially or in various combinations.

A modern coal cleaning plant is a "continuum of technologies rather than one distinct technology".

Coal to be cleaned is usually separated into three size fractions: coarse, intermediate and fine. Preparing coals for more extensive cleaning involves crushing the raw coal feed to smaller size fractions and cleaning the coal particles of smaller and smaller size fractions. Additionally, each fraction can be treated with more specific and sophisticated methods and equipment.

The specific gravity of clean coal is less than that of ash, pyritic sulfur or the other impurities found in raw coal. Most coals have a specific gravity that ranges from 1.12 to 1.70. Ash and other impurities have a wide range of specific gravities: Pyritic sulfur-4.6 to 5.2; gypsum, kaolinite and calcite (typical ash constituents)-2.3, 2.6, and 2.7; and sandstone, clay and shale (typical mining wastes)-2.6. This difference in specific gravity is used by almost all of the commercially-used wet physical coal cleaning technologies to separate larger-sized coal particles (larger than 1/2 mm) from their associated impurities.

These specific gravity-based technologies, however, are usually not effective in cleaning raw fine-size coal particles smaller than 1/2 mm in size. When the size of coal is reduced, the specific surface area of the resulting fine coal particles is increased. Because of this increase in the specific surface area, the separation times are longer for the fine particles.

Six processes are commonly used to clean coal:

Jigs are one of the oldest, and still the most common technology used for washing, separating and concentrating mineral ores. Innovations in design have kept jigs competitive with other cleaning devices. For example, the Batac jig can process finer-sized coals more efficiently than the conventional Baum jig.

Dense media vessels use liquids that are heavier than water to separate coal from its impurities. Particles of coal as small as 1/4 inch in size can be treated with this process.

Dense medium cyclones are used to effect a sharper separation of intermediate sized raw coal material (1/2 inch x 1/2 mm). In this process, the gravity separation of a dense-media mixture is enhanced by centrifugal force.

Hydrocyclones or water-only cyclones also enhance separation by centrifugal force, but they do not use a dense-media mixture. They are now being used more often in new preparation plants as rougher separating devices that decrease the load on downstream cleaning equipment. They are also used to clean very small flotation size coal (-28 mesh).

The concentrating table, a common hydraulic separator, is one of the oldest and most widely used cleaning devices in the United States. It is used especially for processing particles in the 3/8 inch to 0 size range.

The froth flotation process is used to clean fine coal particles of less than 1/2 mm in size. Technologies used to remove the impurities associated with fine coal particles take advantage of the fact that coal, like oil, has surface properties that make it "hydrophobic" or water-hating. The most important of these technologies is froth flotation. In this process, raw fine coal particles are immersed in a water bath, through which air is bubbled. The water-hating coal particles cling to these air bubbles (nearly always with the help of flotation reagents) and can be skimmed off in a surface froth. The heavier coal impurities, which are not so hydrophobic, sink and mix with the water (Hutton and Gould, 1982).

The oil agglomeration process, which is still in the research and development stage, utilizes the same water-hating properties of coal particles, but uses oil droplets instead of flotation reagents. The oil agglomeration process is a wet mechanical beneficiation process that beneficiates the coal by forming microagglomerates or flocs of the coal particles. The process is especially suited for the beneficiation of fine-sized coals and yields the best results when used with other wet mechanical beneficiation processes such as heavy-medium or gravity separation. The process may be used either as an alternative to the froth flotation process or as an additional processing step following froth flotation to recover the very fine-sized (<200-mesh) coal particles that are generally not recovered by the froth flotation process (Khoury, 1981).

### 3. BACKGROUND INFORMATION ON OIL AGGLOMERATION

#### 3.1. Basic Principles

Oil agglomeration, or the so-called spherical agglomeration, is a process of forming large dense agglomerates of considerable integrity from fine particles in liquid suspension by the addition of suitable amounts of a second immiscible liquid (bridging liquid) under appropriate agitation conditions.

The method designed to extract ultrafine particles with an organic phase is based on the following theoretical considerations. When a mixture of two immiscible or partially miscible liquids is shaken, a dispersion of one in the other results; but, in order to attain any degree of stability, a third component is necessary. This third substance need not necessarily be a surface-active agent but can be a finely divided solid. If the two immiscible liquids are a neutral oil and water then, depending on the relative phase volumes of the oil and water, and the type of solid, either an oil in water (O/W) or a water in oil (W/O) dispersion will result (Mellgren and Shergold, 1966).

Young's equation of wetting of solids by liquids, is

$$\gamma_{so} - \gamma_{sw} = \gamma_{wo} \cos \theta$$

where  $\theta$  is the contact angle in the aqueous phase and  $\gamma_{so}$ ,  $\gamma_{sw}$ , and  $\gamma_{wo}$  are the surface free energies at the solid-oil, solid-water and oil-water interfaces, respectively.

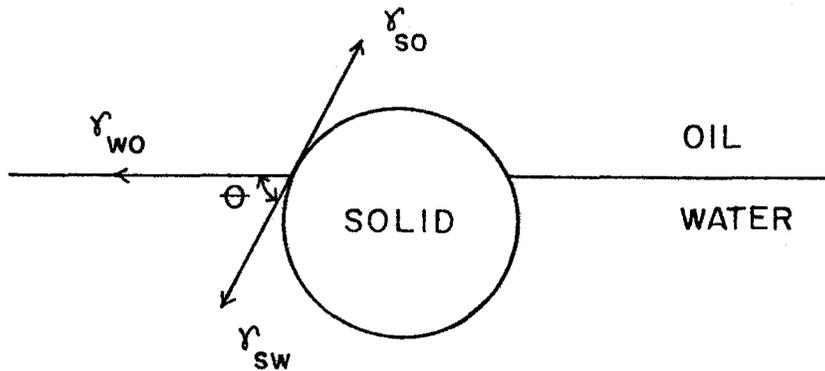


Figure 1. Forces acting on a solid particle at an oil-water interface.

According to Mellgren and Shergold (1966) three situations may occur.

1. If  $\gamma_{so} > \gamma_{wo} + \gamma_{sw}$ , the solid will remain dispersed in the aqueous phase.
2. If  $\gamma_{sw} > \gamma_{wo} + \gamma_{so}$ , the solid will be dispersed in the organic phase.
3. If  $\gamma_{wo} > \gamma_{sw} + \gamma_{so}$ , or if none of the three interfacial tensions is greater than the sum of the other two, the solid particles will concentrate in the water-oil boundary. When the solid collects at the liquid-liquid interface Young's expression can be utilized to give the following results.

1. If  $\gamma_{sw} < \gamma_{so}$ , then  $\cos \theta$  is positive and  $\theta < 90^\circ$ ; and this will result in the major portion of the solid being in the aqueous phase.
2. If  $\gamma_{so} < \gamma_{sw}$ ,  $\cos \theta$  is negative,  $\theta > 90^\circ$ , and the major portion of the particle will be in the organic phase.
3. In the unlikely case when the contact angle is exactly  $90^\circ$ , the particle will be equally wetted by both the organic and aqueous phase.

An important aspect of oil agglomeration is the relative wettability of the components of the suspended solids by the two immiscible liquids. Preferential wetting of oleophillic/hydrophobic particles by oils forms the fundamental basis for the selective agglomeration of one or more components of a complex solids mixture. This selectivity allows naturally hydrophobic coal particles to be wetted by oil and hydrophillic mineral matter to remain in aqueous suspension. In the presence of an adequate amount of oil and sufficient mechanical agitation, the oil-coated coal particles collide with each other and form into agglomerates due to the interfacial tension of the oil and the capillary attraction of the oil bridges between particles.

From a thermodynamic standpoint, the driving force for the wetting by oil and subsequent agglomeration of carbonaceous particles is the reduction in the total surface energy of the system. Thus, the success of the oil agglomeration process depends upon the surface

properties of the solid particles and that of the oil.

The process is most effective if

1. the surface energy of the solid-water interface is high,
2. the surface energy of the solid-oil interface is low,  
while,
3. the surface energy of the oil-water interface is high.

From a process-kinetic standpoint, mechanical agitation is the prime variable affecting the process and is necessary to bring the oil-coated particles into proximity so that the forces responsible for agglomeration may become operative. The extent of mechanical agitation in conjunction with the amount of oil addition determines the rate of formation of agglomerates and their final size. High shear quickly forms agglomerates and reworks them by redispersion and reformation allowing cleaner pellets to form if the hydrophilic particles are removed from the system. The high shear, however, limits the final size of the agglomerates to small diameters (Mehrotra et al., 1983).

The process of agglomerate formation is known to depend on the amount and distribution of the bridging liquid present in the system. Three distinct states of pore filling by bridging liquid have been postulated, namely the pendular, funicular, and capillary states, as presented pictorially in Figure 2. Pendular bonding between particles (Figure 2a) occurs at low levels of bridging liquid and results in loosely flocculated structure which

collapses to a more compact form on addition of sufficient bridging liquid to give the continuous phase required by the funicular state (Figure 2b). At this point the particle network still contains substantial amounts of entrapped suspending medium. Ultimately, the agglomerate pore volume becomes saturated with bridging liquid to give the capillary state (Figure 2c) which is usually considered to represent the most dense agglomerate form (Meadus and Sparks, 1983). Beyond this region the agglomerates exist as pasty lumps; the solids are then essentially dispersed in the bridging liquid (Capes et al., 1977).

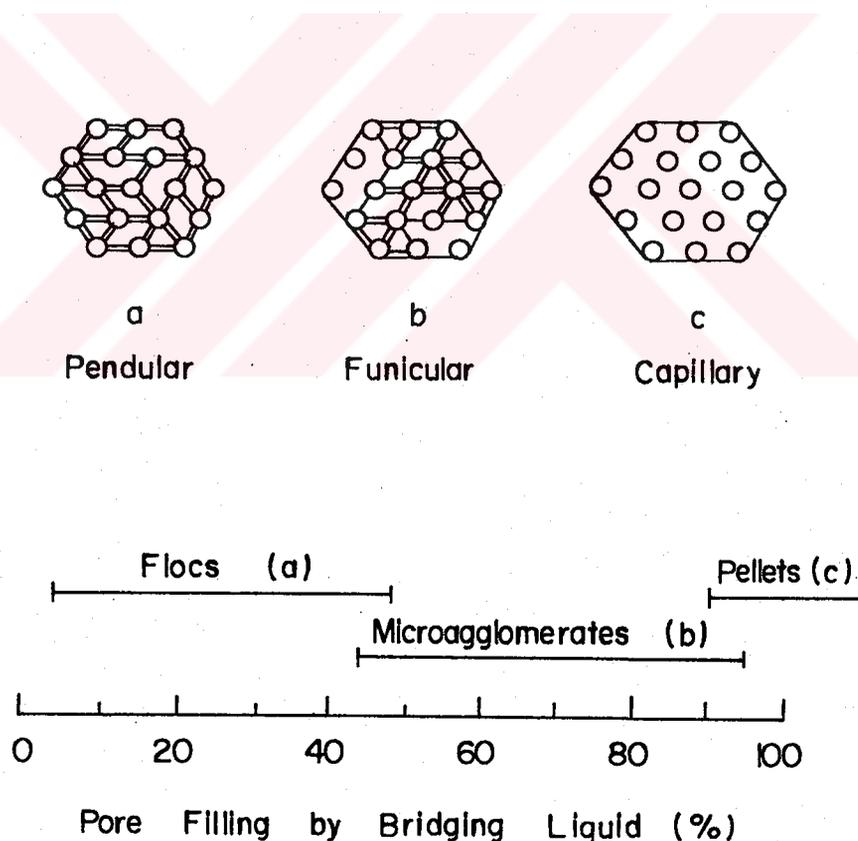


Figure 2. Bridging liquid distribution in agglomerates (Meadus and Sparks, 1983).

Some possible growth mechanisms are given on Figure 3. In the case of coalescence (Figure 3a) two or more agglomerates combine to produce a substantially larger entity. On further agitation or tumbling, the coalesced agglomerates again assume a spherical form. The layering mechanism occurs when free particles become attached to the surface of existing agglomerates (Figure 3b). This is the most controllable form of growth, resulting in optimum particle packing and agglomerate densification. The agitation required to drive the agglomeration process also creates turbulence which can result in agglomerate destruction. Because the smallest agglomerates are the weakest, this group is the most susceptible to breakdown. Broken pieces from this crushing process may be incorporated into larger agglomerates, as shown on Figure 3c. All three growth mechanisms can occur simultaneously, and the preponderance of any one mechanism will depend on the experimental conditions and the characteristics of the material being agglomerated (Capes, 1976).

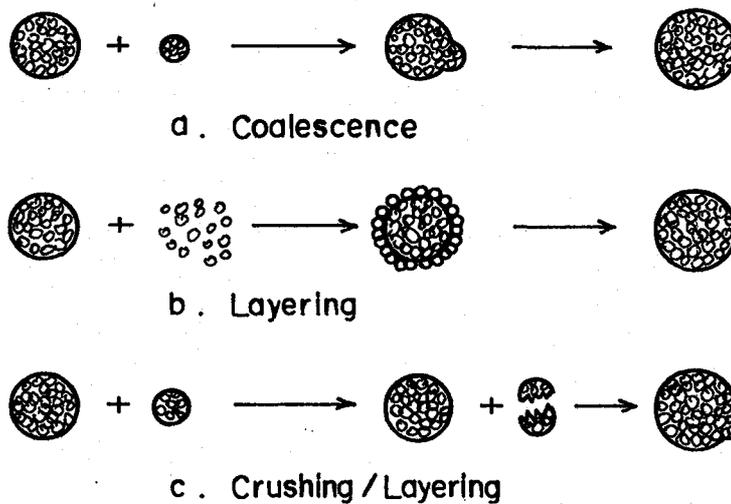


Figure 3. Schematic illustration of agglomerate growth mechanisms (Capes, 1976).

### 3.2. Practical Applications

Oil agglomeration has two entirely separable functions. The more frequently studied is its ability to selectively separate coal from mineral matter, and to beneficiate the coal to a higher grade.

The second function of oil agglomeration is dewatering of coal fines—either to aid conventional dewatering or to replace conventional dewatering and drying entirely.

The most frequent goal of dewatering fine coal is to improve handling and prevent freezing, sticking in bins, or leaking from cars. At the other extreme, dust caused by fine coal handling is a loss, nuisance, and air pollution concern. Oil agglomeration can provide a free flowing, nonfreezing and nondusting product. This aspect of oil agglomeration is of particular value when the proportion of fines is high and some treatment is necessary.

Oil agglomeration may include all the steps shown in Figure 4, but usually only includes the step of mixing oil with the slurry, followed by recovery of the agglomerated product and dewatering on a screen. The final steps shown are used to maximize both grade and hardness of the agglomerated product and recover the more expensive and selective oil for recycle (Mehrotra et al., 1980).

The mixing and recovery steps are common to all the processes, and the distinctions between the processes involve the oils used, the types of mixers used, and the addition of steps to accomplish specific objectives.

The following descriptions of oil agglomeration processes are intended to show the differences between them, although these differences are really smaller than the similarities.

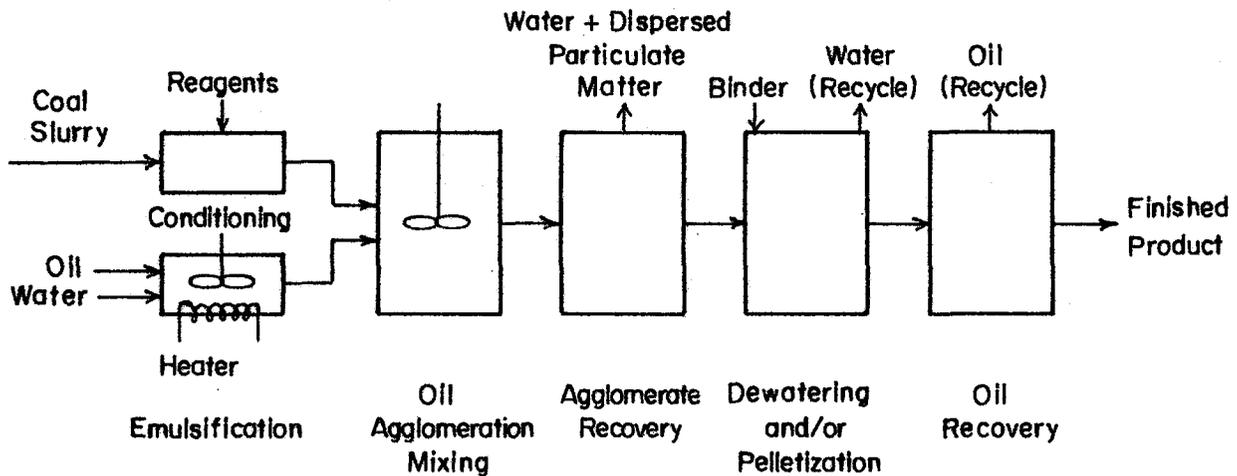


Figure 4. Generalized process flow sheet for oil agglomeration (Mehrotra et al., 1980).

### Trent Process

This is the first oil agglomeration process that was used for coal. In the Trent process, powdered coal, water and about 30% oil (by weight of dry coal) were beaten together to form an amalgam of cleaned coal which was collected on a screen. Because of the higher oil consumption, the Trent process was discontinued after a few years of operation (Capes et al., 1970).

### Convertol Process

In the Convertol process, 3 to 10% oil (by weight of dry coal) was mixed with the slurry under vigorous

agitation and the product was discharged directly to a high speed screen (60 to 80 mesh) centrifuge for dewatering (Sun and McMorris, 1959). The product was a fluffy adhesive mass when wet, but became very dusty when dried and the oil evaporated. The process was not continued, because of the dust problem (Brisse and McMorris, 1958).

### Spherical Agglomeration Process

The most significant difference between spherical agglomeration and Convertol is the degree of compaction and sphericity of the final product. Once the product is highly compacted and spherical, dewatering is simplified. In this process, agglomeration takes place in two steps. First, the coal slurry is agitated with light oil in high shear blenders to generate micro-agglomerates or flocs. Light oils should be preferred in this stage because of their high ash-rejection qualities and better dispersion (Capes and et al., 1971). Next, the micro-agglomerates are dewatered on a screen and then formed into larger pellets on a disc or drum pelletizer. Additional amounts of heavy and cheaper oils are used as a binder in this stage to produce sufficiently strong pellets (Capes et al., 1974).

### Shell Pelletizing Separator

In this equipment, emphasis is placed on the mechanics of mixing the oil phase with the raw coal slurry

in a large multi-bladed reaction vessel. Agglomerates are dewatered by screening (Verschuur and Davis, 1976).

### Olifoc Process

In this process, the fine coal feed is split into two streams using a cyclone to classify it into fine and ultrafine fractions. The ultrafine coal is treated by oil agglomeration and screening, and the fine coal is treated by flotation and filtration (Nicol et al., 1980).

### Selective Agglomeration Process

One of the criticisms of the processes described thus far has been the high energy inputs needed to cause phase separation. Attempts to reduce energy consumption have included using low viscosity oils and preheating the heavy oils. In the selective agglomeration process energy savings are claimed by efficient emulsification of the oil phase (by using a jet whistle emulsifier) prior to its addition to the coal slurry. By including an emulsification step, it has become possible to use heavy oils to reduce the operating costs of the process. It was also claimed that emulsification enhances the oil-particle contact during the agglomeration stage and the use of a high-speed stirrer could be avoided. Emulsification is not energy intensive compared to the energy required if the same oil droplet size is to be obtained during the agglomeration stage. In the selective agglomeration process, the oil agglomerated slurry is discharged onto a sieve bend (Bensley et al., 1977).

## 4. MATERIALS AND EXPERIMENTAL PROCEDURE

### 4.1. Coal Samples

Two kinds of coal samples taken from Zonguldak-Catalağzı coal washery were used in this study:

- i) flotation feed coal fines (below 0.5 mm in size)
- ii) middlings.

Size and ash distribution of the two samples are presented in Table 1 and Table 2.

The middlings sample was screened through 0.5 mm sieve and the oversize was crushed in a roll crusher down to -0.5 mm. In case of the study of the effect of feed size both the flotation feed fines and the middlings were dry ground in a porcelain mill down to 100 mesh and 200 mesh top sizes.

### 4.2. Oils

Two types of oil were used in the experiments. These were kerosene (sp.gr.0.78) and solvent naphta (sp.gr.0.86), a coke oven by-product.

### 4.3. Oil Agglomeration Test Procedure

Bench scale oil agglomeration experiments were carried out in a one-liter cylindrical glass vessel of

9 cm diameter which had two baffles. Agitation was provided by a centrally located, flat-bladed (6 blades, 5 cm diameter and 1 cm width) turbine impeller which operated 1 cm above the bottom of the vessel. Stirring speed was 500 rpm.

Table 1. Size and Ash Distribution of the Flotation Feed Sample.

Size ( $\mu\text{m}$ )	Weight Percent	Ash Percent
+589	8.07	37.15
-589+417	17.67	36.86
-417+295	16.23	37.89
-295+208	9.40	38.51
-208+147	22.37	44.36
-147+104	9.54	50.07
-104+74	5.93	52.71
-74	10.79	53.41
Whole Coal	100.00	42.87

In a typical agglomeration experiment a known amount of coal was mixed with water to make up the required solids concentration. The resulting suspension was then agitated for 5 minutes to provide dispersion of coal and mineral particles and the desired amount of oil phase added by means of a pipette. The slurry was

Table 2. Size and Ash Distribution of the Middlings

Sample

Size ( $\mu\text{m}$ )	Weight Percent	Ash Percent
+6350	3.63	59.22
-6350+4760	6.12	57.58
-4760+3180	14.56	55.60
-3180+2400	9.82	51.85
-2400+1651	15.18	53.33
-1651+1200	10.46	54.11
-1200 +833	12.08	56.81
- 833 +589	8.50	54.63
- 589 +417	5.98	46.75
- 417 +295	3.33	41.20
- 295 +208	1.81	38.03
- 208 +147	3.54	38.13
- 147 +104	1.55	40.97
- 104 +74	1.13	40.90
-74	2.31	42.68
Whole Coal	100.00	52.41

then stirred for 30 minutes to allow for agglomerate growth. The agglomerates were separated on a 65 mesh sieve when 100 mesh top size particles were agglomerated and on a 150 mesh sieve when 200 mesh top size particles were agglomerated. The sieves retained the agglomerated coal while the water and tailings passed through the sieves. The agglomerates were rinsed with water by hand jigging and were dried in an oven at 105°C, weighed and analyzed for ash.

#### 4.4. Flotation Test Procedure

Conventional flotation tests were conducted with a Denver Laboratory Sub-A flotation machine fitted with a one-liter glass cell. The coal-water mixture prepared to desired pulp density was agitated for five minutes. Kerosene was added and the system conditioned for one minute. MIBC (frother) was then added and after one minute conditioning, the air valve was turned fully on and a froth was produced. The froth was skimmed into a tray until a visible collection ceased, and the concentrate and tailings were collected and oven dried at 105°C. The dried products were weighed and analyzed for ash.

#### 4.5. Ash Analysis and Combustible Recovery Calculation

The ash analysis of test products were performed according to T'S Method 330 standard method. About one gram of coal is weighed into a dish and placed in a

cold furnace ventilated by air, the temperature slowly raised to 800°C and maintained at this temperature until constant weight is obtained. The dish is cooled, first in the open air and then in a desiccator, and the mass of ash in the dish determined. The mass of ash, expressed as a percentage of the amount of coal taken, is the ash content.

The recovery of combustibles is calculated from the following formulae :

$$R = \frac{C_A}{C_F} \times 100$$

$$C_A = M_A(1 - A_a)$$

$$C_F = M_F(1 - F_a)$$

where  $M_A$  : weight of agglomerated product

$A_a$  : ash in agglomerated product, percent

$C_A$  : weight of combustibles in agglomerated product

$M_F$  : weight of feed coal

$F_a$  : ash in feed coal, percent

$C_F$  : weight of combustibles in feed coal

$R$  : percent recovery of combustibles.

## 5. RESULTS AND DISCUSSION

A number of batch oil agglomeration tests were performed to study the influence of various variables. Variables studied were the type and concentration of oil, pulp density and fineness of feed. Conventional flotation tests were also conducted to make a comparison between oil agglomeration and flotation test results. The experimental data obtained are given in the appendix, and the results are discussed in the following sections.

### 5.1. Effect of Oil Dosage

The effect of kerosene dosage on the recovery of combustibles and ash in oil-agglomerated coal product is shown in Figure 5 and Figure 6 for the flotation feed samples and Figure 7 and Figure 8 for middlings samples, respectively.

It was observed that a wide range of oil dosages was effective in producing agglomerates. In the lower range of oil loading, only a few pendular bridges can form between the particles and an unconsolidated floc structure is formed. When such flocs are separated on a screen of suitable size, yield is poor because of the poor integrity of the agglomerates. The flocks tend also

to trap inorganic ash components and moisture which drains only slowly by gravity alone. With larger amounts of oil, the chain-like floc structure is replaced by more compact three-dimensional agglomerates. This is the funicular region of interparticle void filling in which the number of oil junctions per particle has increased and pendular bridges have partially coalesed into a network structure. Such agglomerates are more discrete in nature and ash particles are able to drain more easily with the suspending aqueous phase. The agglomerates grow in size and reach a peak of strength and compaction near the capillary region when the void space in the interior of the agglomerates is just filled with oil. Beyond this region, the agglomerates contain too much oil to remain relatively firm. They become soft, paste-like, cohesive lumps in which the solids are essentially dispersed in the bridging liquid.

Fine grinding is necessary in order to obtain lower ash agglomerates. As the fineness of feed increases the ash content of agglomerates decreases and the recovery of combustibles increases. However, finer feeds require more oil. This is due to the fact that with increased fineness of feed, there is better release of mineral matter from coal and there is also an increasing specific surface area. This requires more oil for forming suitable sized agglomerates.

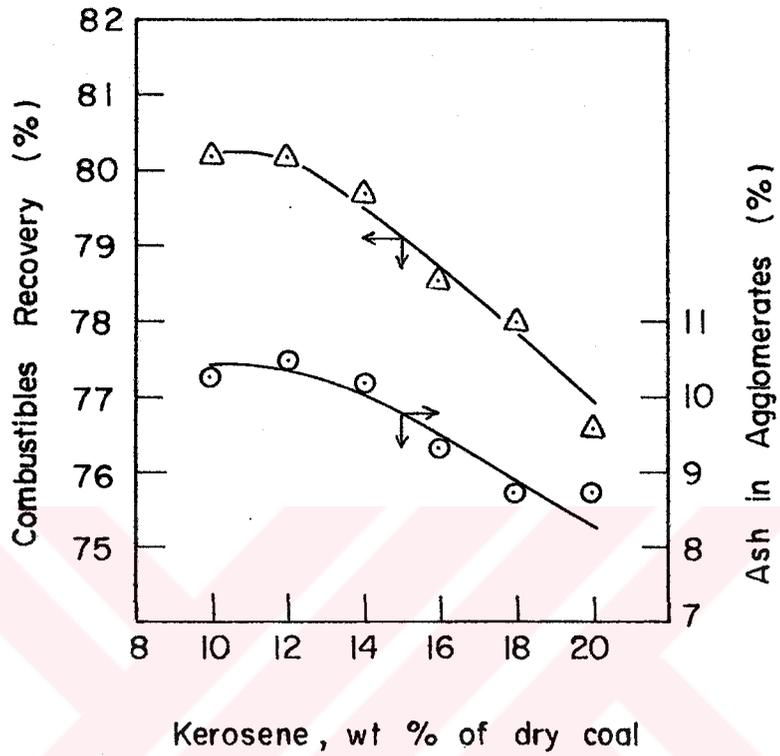


Figure 5. Effect of kerosene dosage on oil agglomeration of -100 mesh ground flotation feed sample.

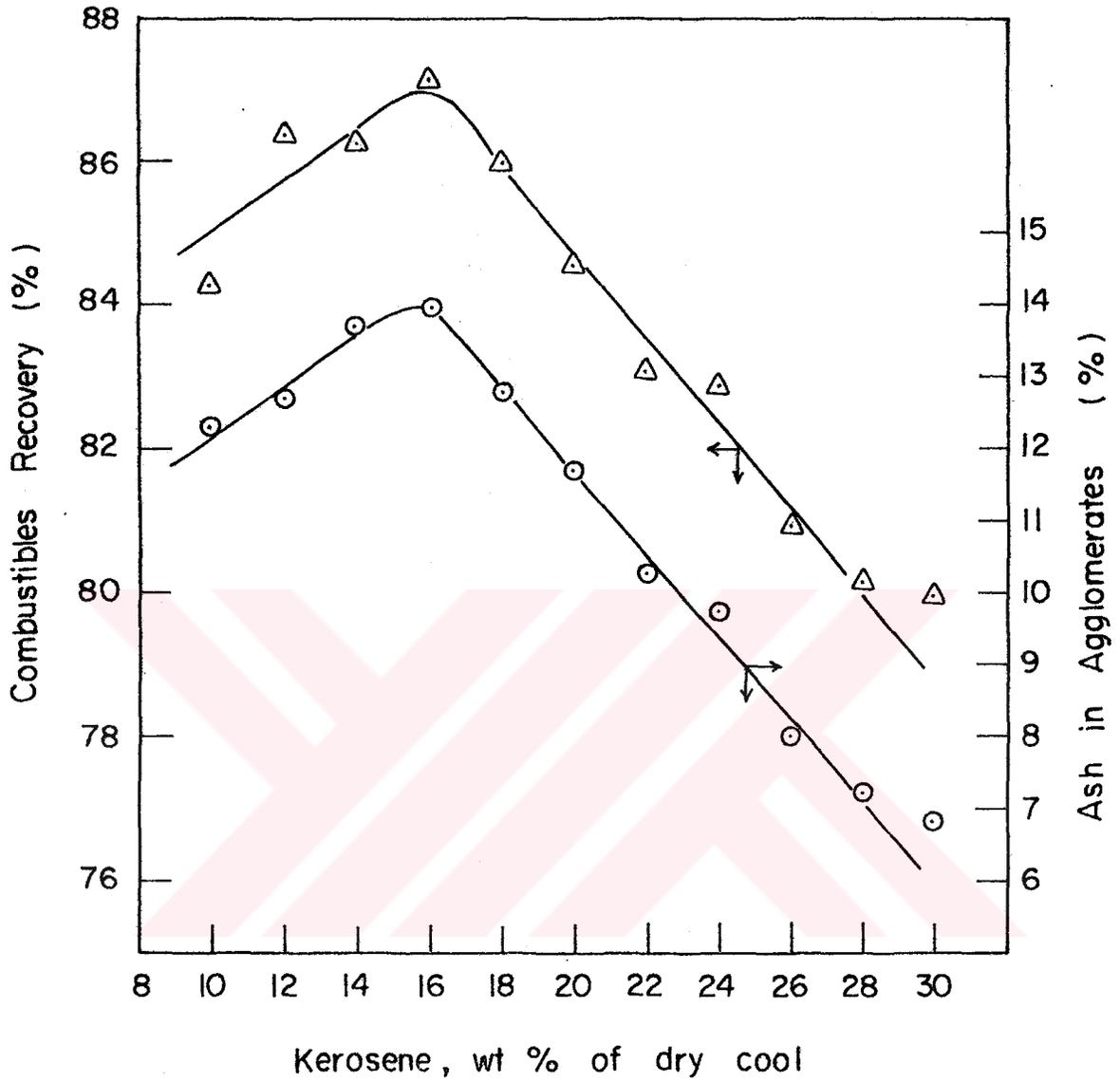


Figure 6. Effect of kerosene dosage on oil agglomeration of -200 mesh ground flotation feed sample.

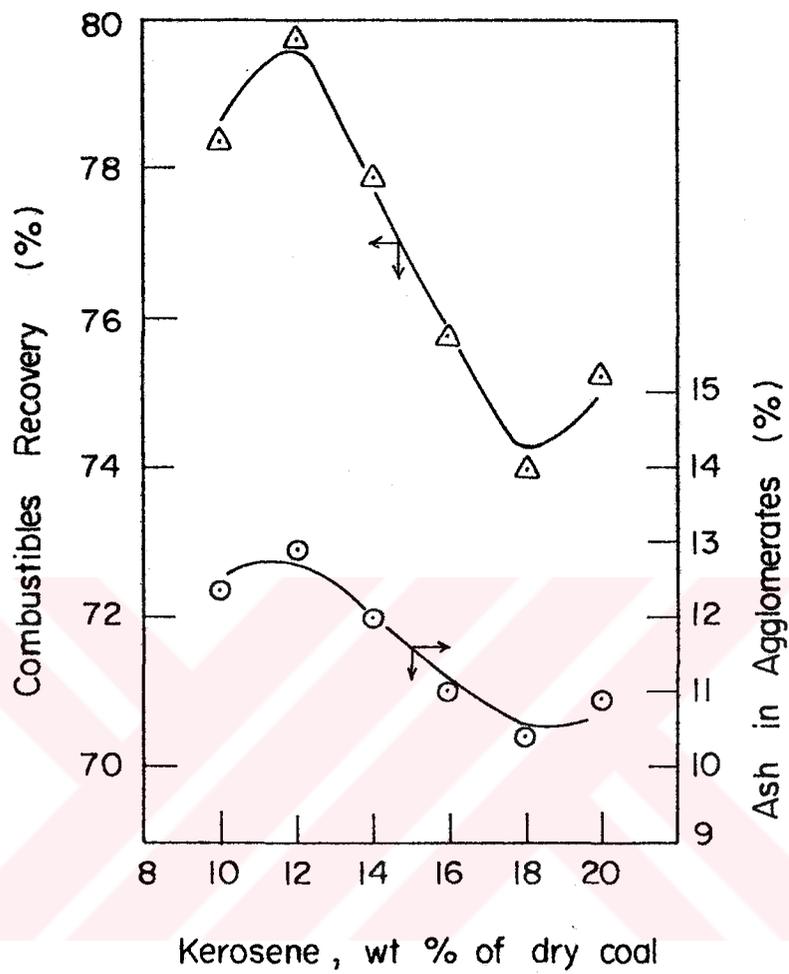


Figure 7. Effect of kerosene dosage on oil agglomeration of -100 mesh ground middlings sample.

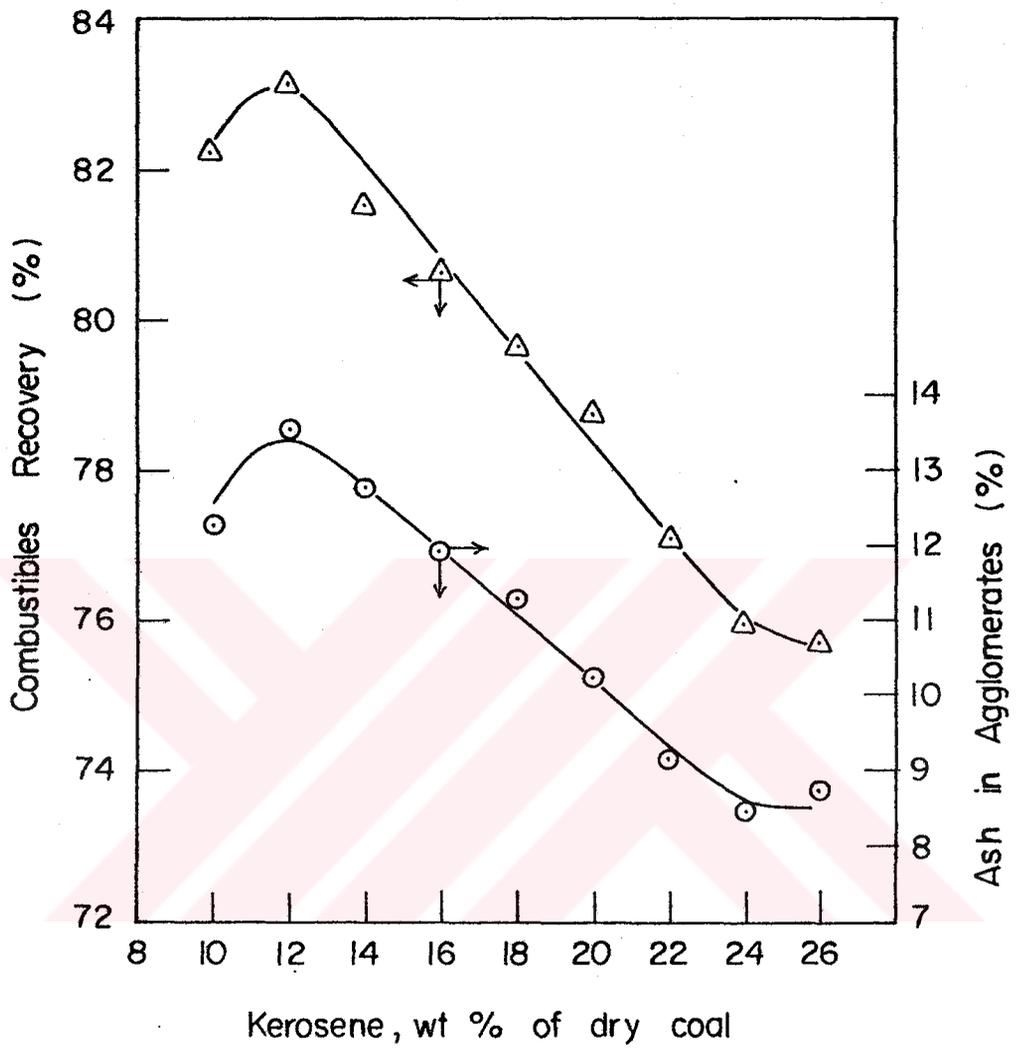


Figure 8. Effect of kerosene dosage on oil agglomeration of -200 mesh ground middlings sample.

## 5.2. Effect of Pulp Density

The effect of pulp density on the recovery of combustibles and ash in oil-agglomerated coal product is shown in Figure 9 and Figure 10 for the flotation feed samples and Figure 11 and Figure 12 for middlings samples, respectively.

Experiences showed that pulp density is not a critical factor in oil agglomeration and slurries up to 50 or 60 percent by weight of solids can be dewatered and deashed to levels comparable to those at lower pulp densities. It is probable that above 40 percent solids, some difficulty in obtaining proper mixing would be experienced.

As the pulp density increased the ash in oil-agglomerated coal product remained constant however, the recovery of combustibles increased because, during stirring, frequency of collisions of oil droplets with coal particles as well as mutual collisions of oil-coated particles increased.

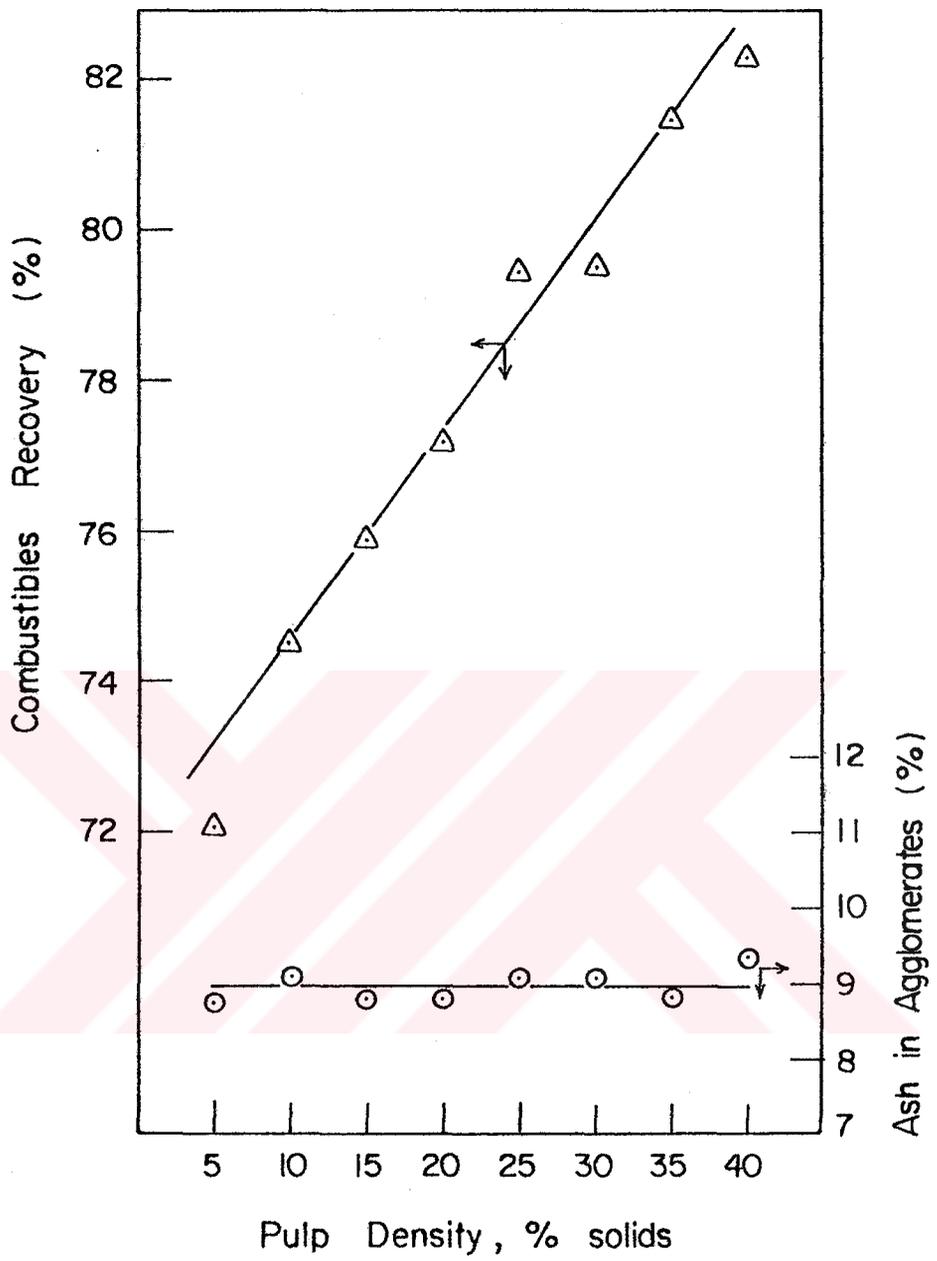


Figure 9. Effect of pulp density on oil agglomeration of -100 mesh ground flotation feed sample.

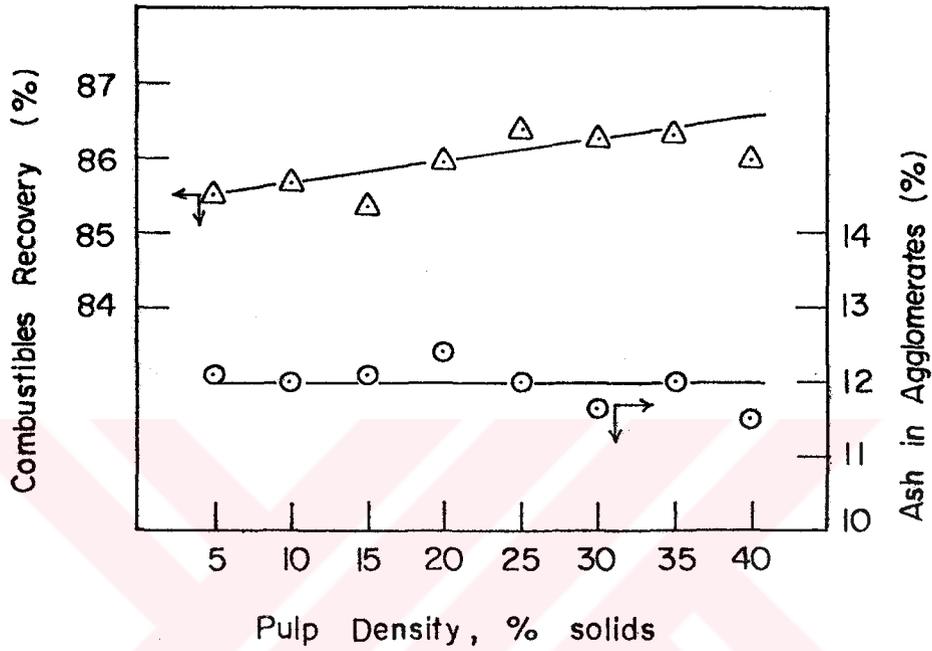


Figure 10. Effect of pulp density on oil agglomeration of -200 mesh ground flotation feed sample.

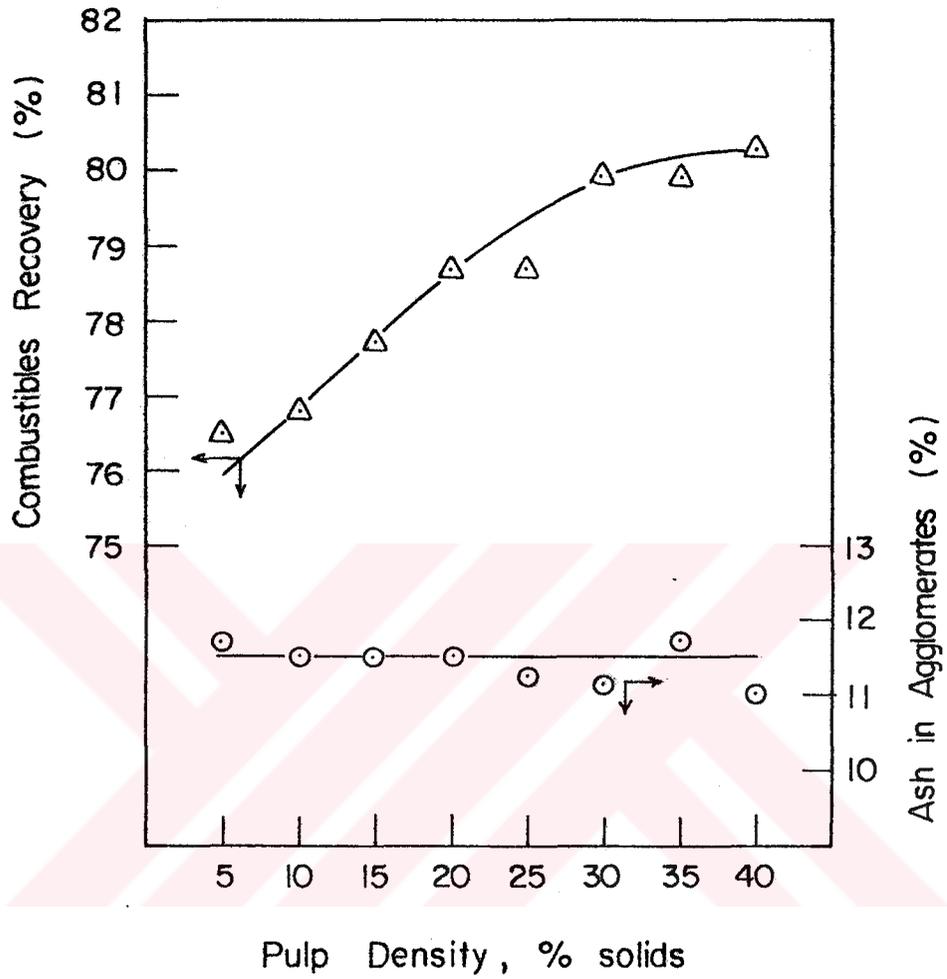


Figure 11. Effect of pulp density on oil agglomeration of -100 mesh ground middlings sample.

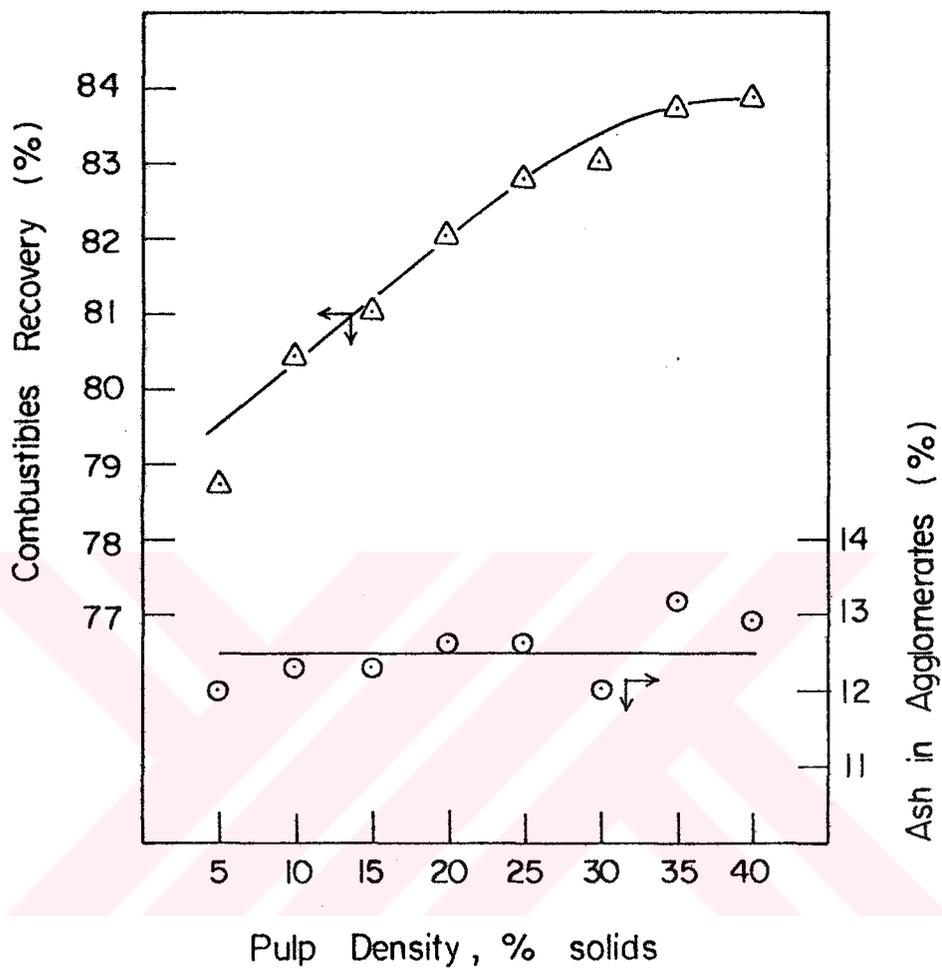


Figure 12. Effect of pulp density on oil agglomeration of -200 mesh ground middlings sample.

### 5.3. Effect of Feed Fineness

Probably the most important characteristic of the oil agglomeration process is its ability to treat a broad range of coal particle sizes. Both the oil agglomeration technique and froth flotation rely on differences in the surface properties of coal and dirt to accomplish separation. Whereas froth flotation becomes less effective where extremely fine coal particles and clay slimes are present, oil agglomeration is able to recover and deash even extreme fines.

The oil agglomeration technique is very efficient in handling extremely fine materials--that is, coals in the -200 mesh range. Modern coal mining techniques have become increasingly mechanized with the result that preparation plants are producing ever-increasing amounts of fines in waste slurries. These fines are difficult to deash and dewater by conventional techniques, but excellent results can be handled by using the oil agglomeration. In addition, the quality of coals being mined is becoming poorer each year. Low-quality coals often mean that ash and sulfur are extremely finely disseminated in the carbon structure, and upgrading can be accomplished only by very fine grinding to liberate the impurities. The oil agglomeration provides an ideal method for reconstituting the coal into a useful product after this fine grinding.

Table 11 and Table 12 (see appendix) show the effect of feed fineness on the recovery of combustibles and ash in oil-agglomerated coal product for flotation feed samples and middlings samples, respectively.

The results suggest that increased fineness of feed sizes lead to increased recovery of less clean concentrates. This is due to the fact that with increased fineness of feed sizes, there is better release of mineral matter from coal and there is also an increasing specific surface area. This requires more oil for forming suitable sized agglomerates.

#### 5.4. Effect of Oil Type

The carbonaceous constituents in coal can readily be agglomerated with many different oils. Heavy, more complex oils--for example, coal tar and bunker C fuel-oil--are not very selective. That is, they not only recover the carbonaceous fraction of the coal slurry but the ash fraction also reports with the agglomerates. By contrast, the light, more refined oils--for example, various solvents, kerosene, and fuel-oil-- are quite selective in recovering the carbonaceous constituents leading to a low-ash product. Apparently the heavier types of oils contain functional groups which are able to condition the ash constituents giving them hydrophobic surfaces and causing them to report with the oil phase during agglomeration.

Table 13 (see appendix) shows a comparison of kerosene and by-product naphtha as coal extracting phases for the oil agglomeration of -100 mesh ground flotation feed sample. Table 13 suggests that at any level of oil, there is no significant difference in ash of products, however with kerosene the recovery of combustibles is higher than that of solvent naphtha.

#### 5.5. Flotation Results

The effect of increasing dosage of oil on recovery of combustibles as well as ash contents of flotation concentrates is shown Figure 13.

In oil agglomeration increasing dosage of oil decreases recovery of combustibles and ash contents of agglomerates (Figure 5). However in the flotation increasing dosage of oil increases both recovery of combustibles and ash contents of concentrates.

Comparison of Figure 5 with Figure 13 suggests that in flotation the recovery of combustibles is higher than that of oil agglomeration process, however in oil agglomeration the ash content of product is lower than that of flotation process.

Oil agglomeration is a more selective process than froth flotation and is less sensitive to variations in feed pulp density. Oil agglomeration has the additional advantage of producing a low moisture content product.

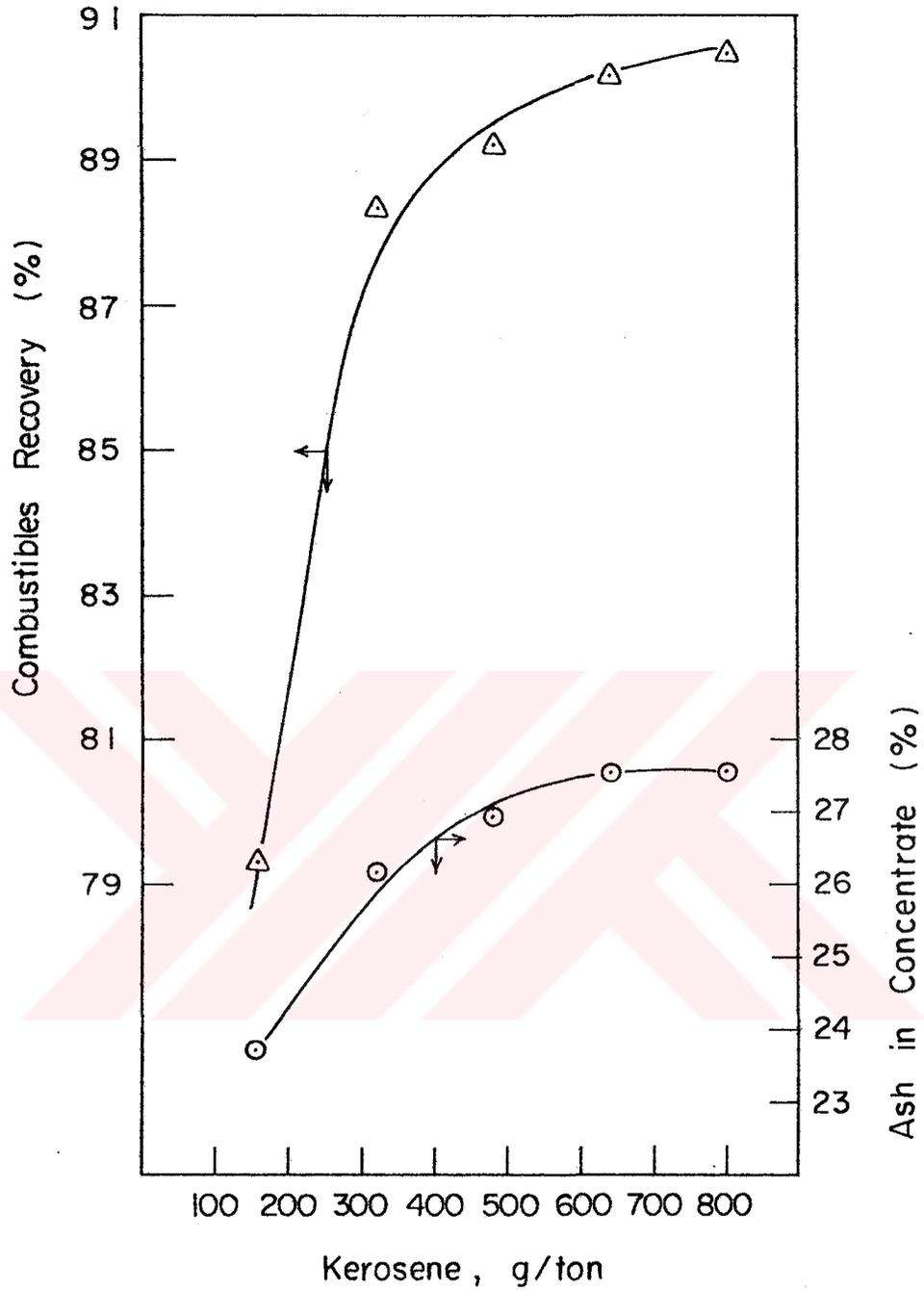


Figure 13. Effect of collector dosage on flotation of -28 mesh flotation feed sample.

## 6. CONCLUSIONS AND RECOMMENDATIONS

1. Dosage of oil controls the ash content of the oil agglomerated coal product. Dosage of oil increases the ash content of agglomerates and the recovery of combustibles decreases.

2. Ash content of agglomerates is not sensitive to variations in pulp density. However, the recovery of combustibles is sensitive to variations in pulp density. The ash content of agglomerates remained constant but the recovery of combustibles increased as the pulp density increased in the range of 5 to 40 percent solids.

3. Fine grinding is necessary in order to obtain lower ash agglomerates. As the feed fineness increases, the ash content of agglomerates decreases and the recovery of combustibles increases; however, the dosage of oil required increases.

4. The type of oil has no significant influence on oil agglomeration efficiency of the coal samples studied.

5. Oil agglomeration is a more selective process than froth flotation process. In flotation, the recovery of combustibles is higher than that of oil agglomeration

process, however in oil agglomeration the ash content of product is lower than that of flotation process.

6. Although two types of light oil (kerosene and naphtha) were investigated in this study, further tests on the use of cheaper and heavier oils (e.g. fuel oil) are recommended.



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APPENDIX

Table 3. Effect of kerosene dosage in oil agglomeration of -100 mesh ground flotation feed sample.

TEST NO	PROCESS VARIABLE	AGGLOMERATED PRODUCT			ASH IN FEED (%)
	KEROSENE (%)	RECOVERY (%)		ASH (%)	
		WEIGHT	COMBUSTIBLE		
1	10	52.2	80.20	10.31	41.62
2	12	50.6	80.17	10.42	43.46
3	14	50.2	79.69	10.14	43.39
4	16	49.2	78.62	9.32	43.25
5	18	49.4	77.98	8.78	42.21
6	20	47.7	76.62	8.75	43.19

OPERATIONAL DATA

Type of coal : Flotation feed  
 Size of feed : -100 mesh  
 Pulp density : 10 %  
 Stirrer speed : 500 rpm  
 Screening : 65 mesh  
 Conditioning time : 5 min.  
 Agglomeration time : 30 min.

Table 4. Effect of kerosene dosage in oil agglomeration of -200 mesh ground flotation feed sample.

TEST NO	PROCESS VARIABLE	AGGLOMERATED PRODUCT			ASH IN FEED (%)
	KEROSENE (%)	RECOVERY (%)		ASH (%)	
		WEIGHT	COMBUSTIBLE		
7	10	55.4	84.33	12.24	42.35
8	12	56.1	86.40	12.74	43.34
9	14	56.5	86.32	13.66	43.49
10	16	58.2	87.11	13.98	42.53
11	18	57.3	86.01	12.80	41.91
12	20	54.0	84.62	11.71	43.66
13	22	53.7	83.12	10.30	42.05
14	24	53.2	82.90	9.75	42.08
15	26	51.0	81.03	8.01	42.10
16	28	49.5	80.20	7.20	42.72
17	30	49.2	80.01	6.80	42.69

OPERATIONAL DATA	
Type of coal	: Flotation feed
Size of feed	: -200 mesh
Pulp density	: 10 %
Stirrer speed	: 500 rpm
Screening	: 150 mesh
Conditioning time:	5 min
Agglomeration time:	30 min

Table 5. Effect of kerosene dosage in oil agglomeration of -100 mesh ground middlings sample.

TEST NO	PROCESS VARIABLE	AGGLOMERATED PRODUCT			ASH IN FEED (%)
	KEROSENE (%)	RECOVERY (%)		ASH (%)	
		WEIGHT	COMBUSTIBLE		
18	10	41.5	78.35	12.40	53.60
19	12	45.0	79.68	12.85	50.78
20	14	41.4	77.87	12.03	53.23
21	16	39.1	75.77	11.01	54.08
22	18	37.7	74.01	10.42	54.37
23	20	37.9	75.23	10.90	55.11

OPERATIONAL DATA	
Type of coal	: Middlings
Size of coal	: -100 mesh
Pulp density	: 10%
Stirrer speed	: 500 rpm
Screening	: 65 mesh
Conditioning time	: 5 min.
Agglomeration time	: 30 min.

Table 6. Effect of kerosene dosage in oil agglomeration of -200 mesh ground middlings sample.

TEST NO	PROCESS VARIABLE	AGGLOMERATED PRODUCT			ASH IN FEED (%)
	KEROSENE (%)	RECOVERY (%)		ASH (%)	
		WEIGHT	COMBUSTIBLE		
24	10	43.9	82.28	12.34	53.23
25	12	45.6	83.17	13.57	52.61
26	14	43.6	81.53	12.79	53.36
27	16	40.4	80.67	11.94	55.90
28	18	41.8	79.68	11.21	53.42
29	20	40.4	78.80	10.24	53.98
30	22	39.7	77.11	9.20	53.25
31	24	39.3	75.99	8.50	52.68
32	26	39.4	75.72	8.70	52.49

OPERATIONAL DATA	
Type of coal	: Middlings
Size of feed	: -200 mesh
Pulp density	: 10 %
Stirrer speed	: 500 rpm
Screening	: 150 mesh
Conditioning time	: 5 min.
Agglomeration time	: 30 min.

Table 7. Effect of pulp density in oil agglomeration of  
-100 mesh ground flotation feed sample.

TEST NO	PROCESS VARIABLE	AGGLOMERATED PRODUCT			ASH IN FEED (%)
	PULP DENSITY (%)	RECOVERY (%)		ASH (%)	
		WEIGHT	COMBUSTIBLE		
33	5	44.1	72.11	8.77	44.21
34	10	47.9	74.50	9.04	41.52
35	15	46.8	75.93	8.80	43.79
36	20	47.9	77.21	8.86	43.46
37	25	49.8	79.42	9.07	42.98
38	30	50.3	79.53	9.01	42.45
39	35	51.2	81.45	8.88	42.72
40	40	51.8	82.31	9.32	42.93

OPERATIONAL DATA	
Type of coal	: Flotation feed
Size of feed	: -100 mesh
Kerosene	: 18 %
Stirrer speed	: 500 rpm
Screening	: 65 mesh
Conditioning time	: 5 min.
Agglomeration time	: 30 min.

Table 8. Effect of pulp density in oil agglomeration of  
-200 mesh ground flotation feed sample.

TEST NO	PROCESS VARIABLE	AGGLOMERATED PRODUCT			ASH IN FEED (%)
	PULP DENSITY (%)	RECOVERY (%)		ASH (%)	
		WEIGHT	COMBUSTIBLE		
41	5	56.1	85.54	12.10	42.35
42	10	56.5	85.69	11.97	41.96
43	15	55.6	85.36	12.14	42.77
44	20	56.1	85.93	12.42	42.82
45	25	54.8	86.40	11.95	44.02
46	30	55.7	86.30	11.61	42.95
47	35	56.2	86.31	11.95	42.67
48	40	55.2	85.99	11.51	43.20

OPERATIONAL DATA	
Type of coal	: Flotation feed
Size of feed	: -200 mesh
Kerosene	: 18 %
Stirrer speed	: 500 rpm
Screening	: 150 mesh
Conditioning time	: 5 min.
Agglomeration time	: 30 min.

Table 9. Effect of pulp density in oil agglomeration of  
-100 mesh ground middlings sample.

TEST NO	PROCESS VARIABLE	AGGLOMERATED PRODUCT			ASH IN FEED (%)
	PULP DENSITY (%)	RECOVERY (%)		ASH (%)	
		WEIGHT	COMBUSTIBLE		
49	5	42.7	76.52	11.71	50.73
50	10	40.2	76.85	11.56	53.74
51	15	41.0	77.77	11.53	53.36
52	20	42.0	78.67	11.55	52.78
53	25	40.5	78.70	11.23	54.32
54	30	40.9	79.89	11.12	54.50
55	35	42.2	79.94	11.73	53.40
56	40	42.2	80.27	11.02	53.22

OPERATIONAL DATA	
Type of coal	: Middlings
Size of feed	: -100 mesh
Kerosene	: 14 %
Stirrer speed	: 500 rpm
Screening	: 65 mesh
Conditioning time	: 5 min.
Agglomeration time	: 30 min.

Table 10. Effect of pulp density in oil agglomeration of  
-200 mesh ground middlings sample.

TEST NO	PROCESS VARIABLE	AGGLOMERATED PRODUCT			ASH IN FEED (%)
	PULP DENSITY (%)	RECOVERY (%)		ASH (%)	
		WEIGHT	COMBUSTIBLE		
57	5	40.5	78.74	12.03	54.75
58	10	42.7	80.44	12.33	53.46
59	15	43.0	81.03	12.34	53.48
60	20	43.9	82.09	12.62	53.27
61	25	43.5	82.80	12.59	54.08
62	30	42.9	83.01	12.08	54.56
63	35	44.8	83.78	13.17	53.57
64	40	44.8	83.89	12.95	53.51

OPERATIONAL DATA	
Type of coal	: Middlings
Size of feed	: -200 mesh
Kerosene	: 14 %
Stirrer speed	: 500 rpm
Screening	: 150 mesh
Conditioning time	: 5 min.
Agglomeration time	: 30 min.

Table 11. Effect of feed fineness in oil agglomeration of flotation feed samples.

Test No	Size of Feed	Oil Dosage (%)	Pulp Density (%)	Ash in Agglomerates (%)	Recovery of Combustibles (%)
4	-100 mesh	16	10	9.32	78.62
10	-200 mesh	16	10	13.98	87.11

Table 12. Effect of feed fineness in oil agglomeration of middlings samples

Test No	Size of Feed	Oil Dosage (%)	Pulp Density (%)	Ash in Agglomerates (%)	Recovery of Combustibles (%)
19	-100 mesh	12	10	12.85	79.68
25	-200 mesh	12	10	13.57	83.17

Table 13. Effect of oil type in oil agglomeration of  
-100 mesh ground flotation feed sample.

TEST NO	PROCESS VARIABLES		AGGLOMERATED PRODUCT			ASH IN FEED (%)
	TYPE OF OIL	OIL DOSAGE (%)	RECOVERY (%)		ASH (%)	
			WEIGHT	COMBUSTIBLE		
3	KEROSENE	14	50.2	79.69	10.14	43.39
5		18	49.4	77.98	8.78	42.21
65	S.NAPHTA	14	48.7	76.55	9.70	42.55
66		18	45.1	72.65	8.69	43.32

OPERATIONAL DATA	
Type of coal	: Flotation feed
Size of feed	: -100 mesh
Pulp density	: 10 %
Stirrer speed	: 500 rpm
Screening	: 65 mesh
Conditioning time	: 5 min.
Agglomeration time	: 30 min.

Table 14. Effect of collector dosage in flotation of  
-28 mesh flotation feed sample.

TEST NO	PROCESS VARIABLE	CONCENTRATE			ASH IN FEED (%)
	COLLECTOR DOSAGE (g/ton)	RECOVERY (%)		ASH (%)	
		WEIGHT	COMBUSTIBLE		
67	160	59.4	79.35	23.70	42.88
68	320	67.9	88.35	26.12	43.22
69	480	69.1	89.24	26.93	43.42
70	640	69.8	90.20	27.50	43.90
71	800	70.8	90.54	27.54	43.34

OPERATIONAL DATA	
Type of coal	: Flotation feed
Size of feed	: -28 mesh
Pulp density	: 10 %
Collector	: Kerosene
Frother-MIBC	: 150 g/ton
Conditioning time	: 7 min.
Flotation time	: 3 min.