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COMPARISON OF IONIZING AND NON-IONIZING COLLECTORS FOR THE FLOTATION OF ZONGULDAK COAL FINES

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ABSTRACT

COMPARISON OF IONIZING AND NON-IONIZING COLLECTORS FOR THE FLOTATION OF ZONGULDAK COAL FINES

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The purpose of this research is to determine the effect of ionizing collectors as an alternative to conventional non-polar collectors for the flotation of high-ash coal fines. The comparison on the basis of yield and ash content of clean coal was done between conventional collectors and ionizing collectors. In flotation experiments, non-polar collectors (kerosene and Pirolizet), ionizing collectors (fatty acids and sulfonates, alone or combined form), frothers (Pine oil, MIBC, Dowfroth 250, diethyl isohexanol) and reagent Accoal E12 were used. Due to the high ash content (46.10 %) of the sample, clean concentrates (below 21 % ash) could not be achieved with non-polar collector-frother combination. Ionizing collectors were found to be good collectors for the production of low ash concentrates although their yields were low (around 50-55 %).

Mixture of fatty acids collectors containing more than 40 % oleic acid, e. g. Pamak 1, Pamak 4 and Tall oil was found better collectors than oleic acid alone and mixing fatty acid and sulfonate collectors was found to be more effective than using them alone. 100-200 g/t Accoal E12 increased the effectiveness of 1200 g/t Pamak 1 (a mixture of fatty acids) importantly. Pine oil and diethyl isohexanol were found better frothers than MIBC when they were used with kerosene.

Key Words: Fine Coal Flotation, Ionizing Collectors, Flotation Promoters

Science Code: 607.02.07

ÖZ

İNCE TANELİ ZONGULDAK KÖMÜRLERİNİN İYONLAŞABİLEN VE İYONLAŞMAYAN TOPLAYICILARLA FLOTASYON ÖZELLİKLERİNİN KARŞILAŞTIRILMASI

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Bu araştırmanın amacı, kül içeriği yüksek ince taneli kömürlerin flotasyonunda iyonlaşabilen toplayıcıları, iyonlaşmayan toplayıcılara alternatif olarak kullanılabilirliğinin tesbitidir. Geleneksel kömür reaktifleri iyonlaşabilen toplayıcılar ile, ürün verimleri ve kül içerikleri açısından karşılaştırılmışlardır. Flotasyon deneylerinde polar olmayan toplayıcılar (kerosene ve Pirolizet), iyonlaşabilen toplayıcılar (yağ asitleri ve sülfonatlar, tek olarak veya birlikte), köpürtücüler (çam yağı, Dowfroth 250, MIBC ve di-etil izohekzanol) ve yardımcı reaktif Accoal E12 kullanılmıştır. Örneğin kül

içeriği çok yüksek (% 46.10) olduğu için geleneksel polar olmayan reaktiflerle % 21 nin altında temiz ürün elde edilememiştir. İyonlaşabilen toplayıcılar ise düşük ürün verimlerine (% 50-55) rağmen, düşük küllü ürün eldesinde iyi toplayıcılar olarak gözükmüşlerdir.

Pamak 1, Pamak 4 ve Tall oil gibi % 40 dan daha fazla oleic acid içeren yağ asitleri toplayıcıların oleic acid' den daha iyi toplayıcı oldukları görülmüştür ve yağ asitleri ve sülfonatların karışım şeklinde kullanımlarının onların tek olarak kullanımlarından daha etkili olduğu bulunmuştur. 100-200 g/t Accoal E12 ilavesi, 1200 g/t Pamak 1'in etkisini önemli ölçüde arttırmaktadır. Çam yağı ve di-etil isohekzanol, kerosenle beraber kullanıldıklarında MIBC den daha iyi netice vermişlerdir.

Anahtar Kelimeler: İnce Kömür Flotasyonu, İyonlaşabilen Toplayıcılar, Promoter

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CHAPTER I

INTRODUCTION

Zonguldak is the main bituminous coal region of Turkey. The bituminous coal reserves of Turkey is around 1.3 billion metric tons. Annual production is around 5 million tons, all of the run-of-mine bituminous coal is treated in four coal preparation plants in Zonguldak area. The largest coal washery in Zonguldak is the main coal washery which receives about 150.000 tons/month from Üzülmez and Kozlu districts. The washery consists of three units; 100-6 mm coals are treated by Baum jigs, 6-0.5 mm coals are washed by feldspar jigs and minus 0.5 mm goes to thickener for thickening and desliming.

Up to 1990, fine coal (minus 0.5 mm size) has been cleaned by flotation so that the ash content of coal was decreased from 45-48 % down to 16-18 % with the yield around 45 %. The product was dried and blended with fine jig concentrate and sold to Ereğli Iron and Steel Plant.

Due to the inefficient operation in the flotation circuit (high ash and low yield of concentrate), the operation was stopped and flowsheet was changed by canceling flotation circuit. Nowadays, after dewatering, the fines are sold to Çatalağzı Thermal Power Station.

The purpose of this research is to determine the effectiveness of ionizing collectors as an alternative to conventional non-polar collectors used previously for the flotation of coal fines. The comparison of flotation results of conventional and ionizing collectors was done with respect to yield and ash content of the concentrates.

CHAPTER II

GENERAL

Coal is a combustible matter resulting from the degradation of vegetable matter, largely in the absence of air. In this natural process of coalification, some non-combustible mineral matter is always associated with the combustible material. Coal preparation or cleaning is the process of removing this non-combustible mineral matter from the coal. Large and intermediate size coals (between 150-0.5 mm) is cleaned by coarse or intermediate gravity preparation method. Fine cleaning (-0.5 mm) is done by shaking table, washing cyclones and flotation (Aplan, 1976).

In 1972, only 13 million tons of raw coal were cleaned by flotation in the United States, and from this 8.4 million tons of clean coal were produced. The percentage of clean coal produced by flotation in Europe (England, West Germany, Poland, USSR, etc.) is substantially higher. Coal flotation has been practiced in Turkey for nearly 40 years. In flotation circuits of Zonguldak Main Washery, kerosene and diethyl isohexanol was added to the pulp as collector and frother, respectively (Özbayoğlu, 1977).

2.1 Natural Floatability and Coal Flotation

Gaudin et al. (1957) have postulated that native floatability results when at least some fracture or cleavage surfaces form without rupture of interatomic bonds other than residual bonds. Solids with native floatability or hydrophobicity, such as coal, show substantial contact angles in distilled water under extremely clean conditions. Although the surfaces of hydrophobic solids have a net hydrophobic character, two additional factors must be considered. First, there may be a significant number of hydrophilic sites on the surface. Second, during size reduction crystal planes, other than those exhibiting a net hydrophobic character, may be exposed. As a sequence particles that might be considered to exhibit native floatability may have significant fraction of their surface consisting of other cleavage planes which do not exhibit a hydrophobic character (Fuerstenau et al, 1985).

Unlike other minerals, coal exhibits a high degree of variation with respect to origin, age, rank, moisture content, degree of weathering, physical and chemical structure, and nature of gangue. As a result, it is difficult to develop a unified flotation strategy from sample to sample. Even samples of the same origin undergo surface chemical changes to different extents on prolonged storage (Yarar, 1988).

Coal's response to flotation varies with carbon content and ash content. The maximum contact angle occurs for low volatile bituminous and semi-anthracite coals. With regard to ash content, Gaudin (1957) has noted that the greater the ash content of a coal, the less hydrophobic is the coal. This is expected since silicates and other ash forming minerals are readily wetted by water.

2.1.1 Impurities Associated with Coal

The mineral matter associated with uncleaned coal is made up of impurities present in coal and shales, clays and rocks which are called as extraneous impurities. The most important constituents are quartzite, calcite, kaolinite, the micas, and gypsum. Sulfur may occur with coal as elemental sulfur, in organic combination or in sulfides and sulfates. Elemental sulfur rarely occurs and organic sulfur cannot be removed by physical cleaning methods. Pyrite which is the most common sulfide mineral in coal may be partly separated by flotation (Brown, 1962).

2.1.2 Effect of Clay Slimes on Coal Flotation

The most important group of minerals in coal are clay minerals which accounts for 60-80 % of total mineral matter associated with coal (Mc Clung et al, 1979). Hussain (1992) found that the clay content of flotation feed accounted around 58 percentage of total mineral matter and the most important clay mineral was kaolinite in Zonguldak bituminous coal. Shales, silts and schists or slates associated with coal as overbreak materials from roof and floor add to the clay content of coal coming to washery for cleaning. The presence of ultrafine clay (less than 2 microns) in flotation causes a loss in recovery of values due either to the presence of slime coatings on the mineral or bubble or to consumption of reagents (Arnold and Aplan, 1986a).

2.1.3 Effect of Weathering and Oxidation on Coal Floatability

Coal can be naturally oxidized as a consequence of weathering. Weathering processes for solid coal involve aerial oxidation of the organic and mineral matter (chemical weathering), microbial oxidation of pyrite (biological weathering) and changes in moisture content which may result in a significant slackening or particle size degradation (physical weathering). Coal rank, particle size, the presence of oxygen and its diffusion coefficient, moisture levels and temperature influence the rate of coal weathering. The process can take place in shallow seems exposed to air and weather conditions throughout geological periods or it can occur in the short time between mining and utilization (Garcia et al, 1991). Deleterious effect of coal oxidation on flotation can be cured by increasing the amount of fuel oil collector. Seriously oxidized coals of very low rank will require large amounts of oil or, preferably, the use of a specific collector (Aplan, 1993).

It is thought that the presence of the reactive oxygen group, such as those of humic acid that form upon oxidation, are the major cause of this recovery loss (Arnold and Aplan 1986a). Wen and Sun (1977) have shown that highly oxidized coal exhibits a large negative zeta potential with essentially no isoelectric point. They also found that a humic extract from an oxidized bituminous coal exhibited this behavior as well.

2.1.4 Flotation pH of Coals

Working with high volatile A rank bituminous (hvAb) coal, Zimmerman (1948) found a roughly parabolic, pH-recovery relationship. The optimum recovery was near pH 7-8. It dropped off sharply below pH 4 and above pH 9. Working with Australian coal, Burdon (1962) found optimum pH for coal recovery to be pH 6 to 8. Working with Zonguldak coals, Sarıkaya (1988) obtained optimum recovery at pH 7.4.

More recent work conducted by Xu and Aplan (1993) showed that high volatile C rank bituminous (hvCb) and medium volatile C rank bituminous (mvCb) coals float well over a broad pH range from pH 3-11, commonly around pH 7-8 as an optimum. As the coal rank is progressively reduced, the amount of oil required for flotation increases, while the region of maximum floatability is progressively reduced to pH 4.

2.2 Reagents Used in Coal Flotation

Many coals may be easily floated with only a frother, e.g. pine oil due to their inherent hydrophobicity. For monohydric aliphatic alcohols and monocarboxylic acids to be good frothers, the molecule contain between five and ten carbon atoms, with an optimum of eight. A second reagent, such as non-polar oil, e.g. kerosene is often necessary to increase the yield of flotation. Kerosene is stated as the most effective collector for coal by Brown (1962). But Onlin and Aplan (1987), based on an extensive study of the flotation of a broad range of coal ranks and oxidized coals, concluded that No:2 fuel oil was the best collector, of nearly 175 system studied, on the basis of effectiveness, convenience and cost. Other collectors for flotation of fine coal are creosols, and cresylic acids. Many commercial flotation agents have both frothing and collecting properties, and others have either an activating or depressant effect, depending on concentration. Some heteropolar reagents not only stabilize the froth but also affect the emulsification and spreading of non-polar reagents, disperse fine clay, and are adsorbed by the coal and change the hydrophobicity of the surface (Brown, 1962).

The use of ionizing collectors is considered as a scientific interest in coal flotation. Hussain (1992), working with Zonguldak coals, used Pamak 4 (a combination of fatty acids) as collector and obtained better results than kerosene-MIBC combination and Sablik (1990), working with Poland coals used FK (a collector containing no aliphatic hydrocarbons and being simply a certain kind of alkyloarylic hydrocarbons) as collector and obtained better results than using apolar collector/aliphatic hydrocarbons.

Baarson et al. (1962) stated that fatty acids are sometimes added in the form of water emulsions in which an emulsifying agent such as a water soluble petroleum sulfonate is used. Booth and Freyberger (1962) stated that some sulfonates are employed as emulsifiers for hydrocarbon oils and insoluble anionic collectors such as the fatty acids and tall oils.

Aplan and Arnold (1991) found dodecane, tricresyl phosphate, tributyl phosphate and some phenols, carboxylic acids and amines better collectors for low-rank and oxidized coals. Crozier (1992) stated that tall oil fatty acids are by far the most consumed organic reagents in non-metallic ore flotation. They contain rosin acids, which are frothers and normally do not act as selective collectors.

2.2.1 Reagent Consumption

Amount of reagent required for coal flotation increases logarithmically as the % C decreases linearly (Xu and Aplan, 1993). Gutierrez and Aplan (1984) have noted a semi-logarithmic relationship between the fuel oil consumption and the % C. Brown (1962) noted a similar correlation. High rank bituminous coal, unless oxidized, will often require no oily collector, merely a frother. The quantity of oil used will increase as the rank decreases. Yarar (1988) stated that for freshly ground coal 50-100 g/t of pine oil acts as both collector and frother. On a practical basis, coals of hvAb require less than 500 g/t fuel oil as collector for their flotation. For hvBb and hvCb coals 500-5000 g/t fuel oil collector are used, while subbituminous and lignite coals require more than 5000 g/t. Some of these latter coals may require more than 2500 g/t oil (Aplan, 1993).

In flotation system, when the bubble and the particle merge, penetration of the collector film by that of the frother occurs, with consequent great stabilization of the solid-liquid-air contact. This "unlocking" effect does not occur well if the concentration of the collector is so high that a complete monolayer is formed at the solid-liquid interface, since penetration by the frothing agent is then inhibited (Adamson, !967). Shaw (1970) stated that bubble adhesion to mineral is maximum when there is only 5-15 percent monolayer coverage by the collector oil and decrease with further coverage.

2.2.2 Flotation Promoters

To obtain high coal flotation recoveries, it is important not only have a significant amount and proper kind of hydrocarbon oil collector, but to ensure that the collector or collector/frother system is efficiently conditioned or absorbed onto the coal particle. In other words, if the oily collector/frother is insufficiently dispersed, some coal particles are coated excessively as others are starved. Generally, if more oil is used to counteract this, it may tend to float more ash particles. When the oil is effectively dispersed, economic usage of reagent can be obtained because the finer droplet size produce more collisions, on average, between coal particles and oil droplets.

The use of some surface active agents, or "promoters" can help to increase the adsorption of oily collectors onto the coal surface, e.g. the adsorption of Tensatile DA 120, a solution of an ionic alkyl sulfate with about 8 carbon atoms in the chain; and sodium dodesil sulfate has been investigated, and found that the adsorption of reagent at the coal-water interface was insufficient to make flotation possible in the absence of non-polar oil but did facilitate the spreading of non-polar oil and improve flotation recovery. The adsorption at the water-oil interface also made spreading easier (Brown, 1962).

Sablik (1990) used some polar promoters such as MDB (meleate dibutyl) organic acid butyl ester, ADO organic acid octyl ester, SDO organic acid octyl ester, EKN acylopolyglycoether (saturated acid radical), EKT acylopolyglycoether (unsaturated acid radical) successfully in flotation of bituminous Poland coals and concluded that these promoters increased flotation response of various type of coal and improved concentrate combustible matter recovery. Working with US coals flotation, Scanlon et al (1983) used Accoal 4433 (a promoter of American Cyanamid Company) and kerosene combination and obtained a 14 % increase in coal recovery for the same ash content in clean coal for one plant. They also obtained a 15 % increase in coal recovery with a cost reduction of 0.40-0.09 $ per ton of clean coal for another plant when compared with hydrocarbon oil-frother combination.

Laskowski (1986), working with different promoters, including EKT and Accoal 4433, found that promoters improve flotation of the coals characterized by difficult floatability. Their use seems to be necessary in the flotation of very difficult-to-float coals. However, coals with intermediate floatability float quite satisfactorily if the reagents are properly emulsified.

2.2.3 Surfactant Activity and Emulsification

The ability of reagents to alter the liquid-vapor interfacial tension, of aqueous solutions is called surface activity (Yarar, 1988). Several approach was developed to correlate the chemical structure of surfactant molecules with their surface activity through some quantitative relationship. A successful attempt to quantitatively correlate surfactant structures with their effectiveness as emulsifiers was the Hydrophile-Lypophile Balance (HLB) system. HLB system quantitatively correlate surfactant structure with their effectiveness as emulsifiers. Laskowski (1986) stated that an emulsified collector provides much higher coal recoveries which otherwise cannot be achieved even after very lengthy conditioning. Horsley and Smith (1951) found that emulsifying the fuel oil with a frother or a surfactant will often lead to reduced reagent requirements. Moxon et al (1986) and Chander et al (1994) found that emulsifiers generally increase coal flotation yields by dispersing the collector into finely divided droplets throughout the flotation pulp.

The use of surfactant mixtures can become complicated by the fact that such mixture often produce more stable emulsions than a single surfactant with the same nominal HLB number. The HLB of the mixture is assumed to be an algebraic mean of the HLB's of the components.

$$HLB_{mix} = f_A x HLB + (1-f_A) HLB_B$$

where f_A is the weight fraction of surfactant A in the mixture.

Because the role of interfacial layer in emulsion stabilization, it is often found that a mixture of surfactant with widely differing solubility properties will produce emulsions with enhanced stability. Finally, it is usually safe to say that the more polar the oil phase the more polar will be the surfactant required to provide optimum emulsification and stability.

CHAPTER III

EXPERIMENTAL PROCEDURES AND EQUIPMENT USED

3.1 Sample Preparation

Fine coal was taken by automatic sampler after they pass through the 0.5 mm filter. They were allowed to settle for 8 hours, then decanted and dried in an oven at 60 °C. Dried coal material was divided into 100 g samples by coning and quartering method, and then kept in double plastic bag to avoid any oxidation. Because even a modest oxidation is very detrimental to coal flotation (Aplan, 1976).

3.2 Proximate Analysis

Proximate analysis were carried out to determine ash, volatile matter and fixed carbon content of dried coal. Ash analysis and volatile matter analysis were carried out according to ASTM D 3174-73 and ASTM D

3175-89, respectively. Heraus muffle furnace was used for both analysis. Fixed carbon analysis is the resultant of the summation of percentage ash and volatile matter subtracted from 100. Namely,

Fixed Carbon, % = 100 - (ash, % + volatile matter, %)

3.3 Flotation Equipment and Reagents Used in Flotation Tests

A Wemco Sub-A laboratory flotation machine with 1 liter cell was used for the flotation experiments. A potable pH meter was used for pH control.

3.3.1 Collectors/Promoters

Ionizing and non-ionizing collectors used throughout the tests are;

Kerosene; a non-polar hydrocarbon collector from Shell CO.

Pirolizet; a non-polar hydrocarbon collector + frother from Poland.

Pamak 1; an ionizing fatty acid collector (47.4 % oleic acid + 43.6 % linoleic acid + 1.4 % rosin oil) of Hercules Inc.

Pamak 4; an ionizing fatty acid collector (46.7 % oleic acid + 41.2 % linoleic acid + 4 % rosin acid) of Hercules Inc.

Tall Oil; an ionizing fatty acid collector (40 % oleic acid + 30 % linoleic acid + 30 % rosin acids) of Denver CO.

Oleic Acid; an ionizing fatty acid collector of Merck.

Aeropromoter 801 (AP 801); an anionic petroleum sulfonate promoter of American Cyanamid.

Aeropromoter 825 (AP 825); an anionic petroleum sulfonate promoter of American Cyanamid.

Aeropromoter 848 (AP 848); a synthetic, anionic, petroleum sulfonate promoter designed to function as fatty acid "booster" from American Cyanamid.

Accoal E12; a coal promoter of American Cyanamid.

Since solubilities of fatty acid collectors in water are very low, they were added directly into the pulp, similar to kerosene and frothers. Since the solubilities of sulfonate type promoters in water is high, they were added into pulp as their solution in water when they were used as principle collector. When a combination of fatty acid and sulfonate type collector was tested, they were premixed to obtain an efficient emulsion.

3.3.2 Frothers

Pine Oil (PO); consists largely of terpene alcohols of which alpha-terpineol is major constituent.

Methyl isobuthyl Carbinol (MIBC); partially soluble aliphatic alcoholic frother.

Diethyl isohexanaol (DEH); partially soluble aliphatic alcoholic frother.

Dowfroth 250 (DF 250); a completely water soluble methoxy polypropylene glycol of Dow Chemical Company.

Other reagents are 1 % NaOH solution and 10 % HCl solution used as pH regulators.

3.4 Flotation Procedure

Flotation experiments were carried out with a pulp density of approximately 10 percent solids by weight. For each flotation test, 100 g sample was used. After 30 seconds conditioning, the pulp pH was controlled and flotation collector was added, and then the pulp was conditioned for two minutes. Finally, the frother was added and conditioned for one minute prior to aeration. Froth was collected manually for three minutes. The experiments were carried out at original pH 8.0, unless otherwise mentioned.

Tap water was used throughout the experiments. Arnold and Aplan (1986b) found flotation of coal lower in distilled water than tap water. Impeller speed was kept constant as 1350 rpm. Water soluble collectors were added into the pulp as 10 % solution.

CHAPTER IV

EXPERIMENTAL RESULTS AND DISCUSSION

4.1 Proximate Analysis of Coal Fines

The ash content of fine coal of Zonguldak shows variation between 40 to 55 % by weight during the plant operation. The ash content of the sample taken from the Zonguldak main coal washery was 46.10 %, as seen in Table 1 given in Appendix. Hussain (1992) found that Zonguldak bituminous coal contains kaolinite, illite and chlorite in addition to other minerals such as quartz, muscovite, serpentine, calcite, sepiolite and feldspar. Kaolinite was dominant clay mineral in flotation feed. Arnold and Aplan (1986a) stated that kaolinite and illite contaminate the floated clean coal largely by water carry over mechanism with water. Mishra (1978) said that the presence of ultrafine clay or other slime particles may inhibit coal froth flotation.

4.2 Flotation Tests

Single stage direct flotation test was carried out throughout the experimental works. First, conventional kerosene-frother combination and Accoal E12 addition to kerosene were tested, then other non-ionizing collector (Pirolizet) and ionizing collectors (fatty acids and sulfonates) were tested. The results of the flotation experiments are summarized in Table 2 to 18 and given in Appendix.

4.2.1 Flotation with Non-Ionizing Collectors

4.2.1.1 Flotation with Kerosene and Pine Oil Combination

Up to 1990, fines of Zonguldak coal was floated with kerosene and diethyl isohexanol combination. Kerosene was used also as a conventional collector in this study. The effect of kerosene on the yield and ash content of coal was determined by using a constant amount (80 g/t) of pine oil. The results of the tests which were shown in Table 2 are plotted in Figure 1.

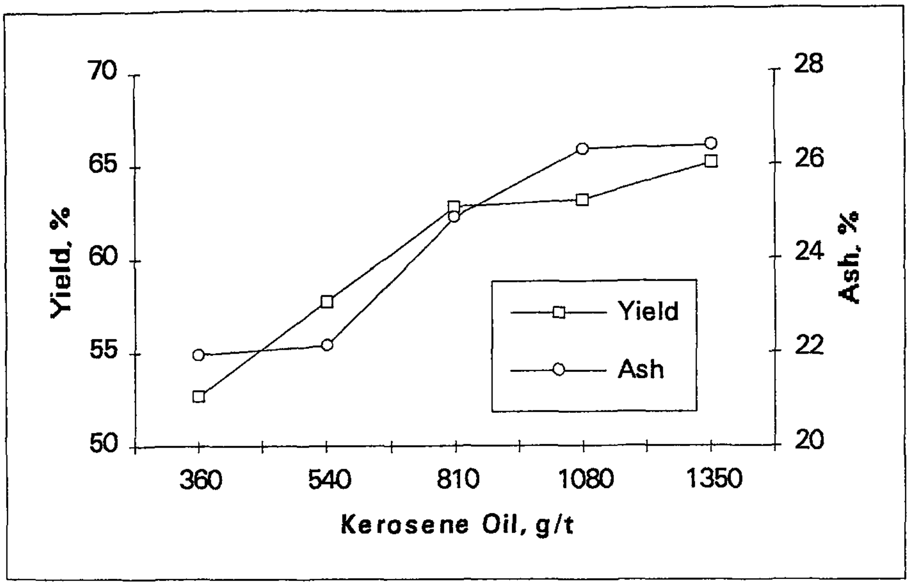

Figure 1. Effect of kerosene Amount

From Figure 1 it is seen that the ash content and yield of concentrate were increased with increasing collector consumption at constant pine oil amount (80 g/t). Since our aim is to obtain a clean coal from high ash Zonguldak coal fines, the concentrate having 22.14 % ash and 57.74 % yield, which was produced by the addition of 540 g/t kerosene, was assumed as an acceptable result.

In order to see the effect of pine oil on the yield and ash content of clean coal floated with kerosene, pine oil was used between 40 and 160 g/t and kerosene amount was kept constant as 540 g/t. The results tabulated in Table 3 are plotted in Figure 2.

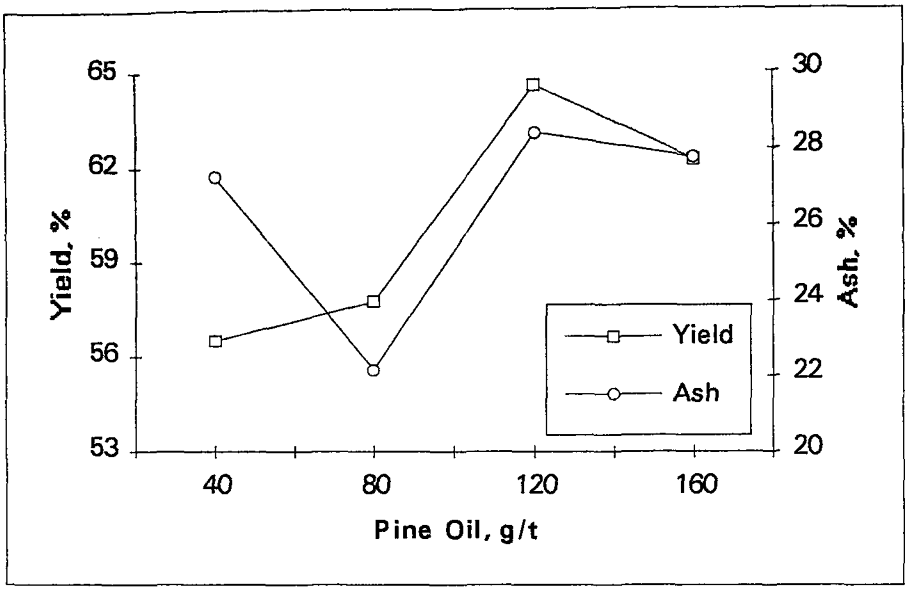

Figure 2. Effect of Pine Oil Amount

As it is seen from Figure 2, the cleanest concentrate was obtained by 80 g/t pine oil addition. At lower and higher consumptions of pine oil, the ash content of the coal increased. At lower frother concentrations, slime particles in the froth were dominant as the air bubble stability may not be sufficient to carry coal particles. At 80 g/t pine oil addition, the strength of the air bubble increased and more coal particles attached on it, resulting an increase in the yield. Higher addition of frother enhanced the physical attachment of the gangue particles to air bubbles, therefore, both yield and ash contents of clean coal increased.

4.2.1.2 Flotation with Kerosene and Accoal E12

As Accoal E12 has the frothing properties, additional frother was not used. Kerosene amount was kept constant as 540 g/t, while Accoal E12 was used between 100 and 500 g/t. The results of tests given in Table 4 are plotted in Figure 3.

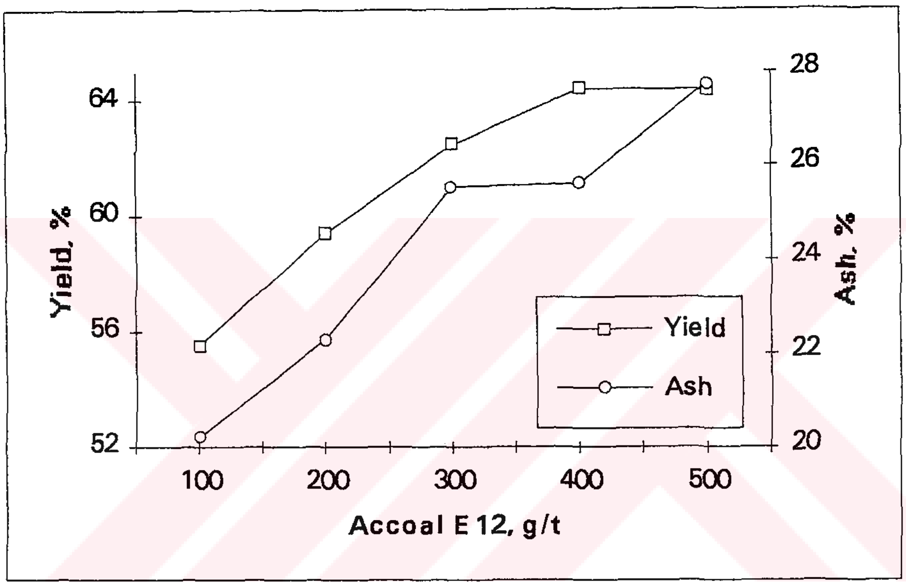

Figure 3. Effect of Accoal E12 Addition

It is seen from Figure 3 that at 100 and 200 g/t promoter additions, clean concentrates with 20.21 % and 22.30 % ash contents, respectively were obtained with acceptable yields (55.52 % and 59.39 %, respectively). When Figure 1 and Figure 3 were compared, the results with

Accoal E12 seems to be better in the terms of yield. But , when the Accoal E12 consumption was increased over 200 g/t, ash contents of the concentrates increased sharply. Working with US coals, Scanlon et al (1983) used about 800 g/t kerosene and 120 g/t Accoal 4433 combination and achieved better results than kerosene-frother combination.

4.2.1.3 Flotation with Different Frothers

Four different type frothers were used during the experiments. The frothers tested are Pine oil (PO), Diethyl isohexanol (DEH), Dowfroth 250 (DF 250), and Methyl Isobutyl Carbinol (MIBC). The amount of frother and kerosene were kept constant as 60 g/t and 630 g/t, respectively. The results of the flotation tests given in Table 5 are plotted in Figure 4.

As it is seen from Table 5, similar concentrates (with about 60 % yield and 22.5 % ash) were obtained with pine oil and diethyl isohexanol. The highest yield was achieved with Dowfroth 250 as 63.34 %, but the ash content was 25.62 % which is higher than the others. As a result, pine oil and diethyl isohexanol are good frother for high ash Zonguldak coal fines.

Sarıkaya (1988) also found pine oil as better frother than MIBC for oxidized Zonguldak coal.

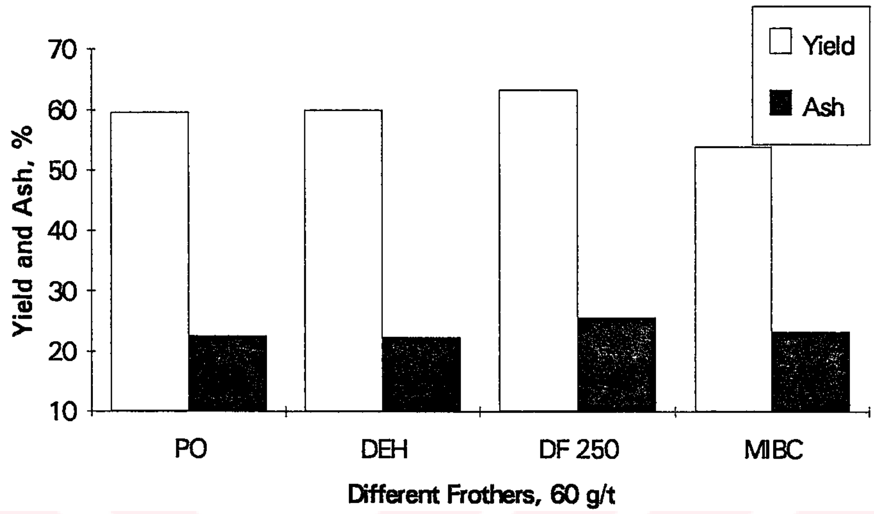

Figure 4. Flotation with Different Frothers

4.2.1.4 Flotation with Pine Oil Alone

Sun (1954) found that the best frothing conditions for pine oil were at neutral or, preferably, alkaline pH values. Additional collector was not used because pine oil, a mixture of terpene alcohols and hydrocarbons acts as a frother/collector system similar to alcohol/kerosene (Read et al., 1989). The results of the tests given in Table 6 are plotted in Figure 5.

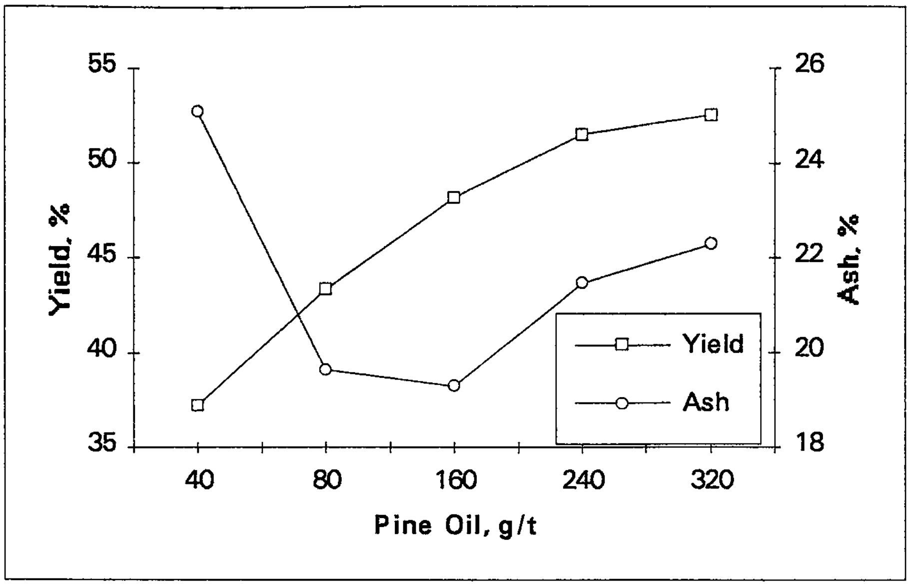

Figure 5. Flotation with only Pine Oil

As it is seen from Figure 5, the concentrates with the highest ash (25.09 %) and least yield (37.22 %) was obtained with 40 g/t pine oil addition. Arnold and Aplan (1986a) stated that kaolinite and illite contaminate the floated coal by carry over mechanism. On the other hand, the strength of the air bubble was so weak due to the insufficient addition of frother that, coal particles couldn't be carried to the surface, therefore resulted in low yield. The same trend was observed in Figure 2. The increase in pine oil addition, up to 160 g/t, strengthened the air bubbles and hence carried coal fines to the surface resulting an increase in yield. Although the yield was low as 48.17 %, the ash content of concentrate decreased to 19.28 % which was the lowest ash content achieved in flotation test with the use of kerosene-frother combination or frother alone.

Addition of pine oil above 160 g/t resulted an increase in the entrapment of inorganic impurities to air bubbles, so both yield and ash contents of the concentrates increased. The usage of pine oil alone in coal flotation has been known since long time. Yarar (1988) stated that for the flotation of freshly ground coal, 50-100 g/t of pine oil acts as both collector and frother.

Although using pine oil alone resulted in low ash in concentrates, when Figure 1 and 5 are compared, it can be seen that, for the same ash content (e. g., 22 %), in clean coal the yield of concentrate produced with kerosene-pine oil combination is 57.74 % while with pine oil alone, it is only 52.56 %. In other words, the yield achieved with the use of pine oil alone is 5 % lower. As Zimmerman (1968) stated, the addition of a collector will increase the response of coal to froth flotation.

4.2.1.5 Flotation with Pirolizet

Pirolizet, a Poland coal reagent, was used both as collector and frother. The results were tabulated in Table 7 and are plotted in Figure 6.

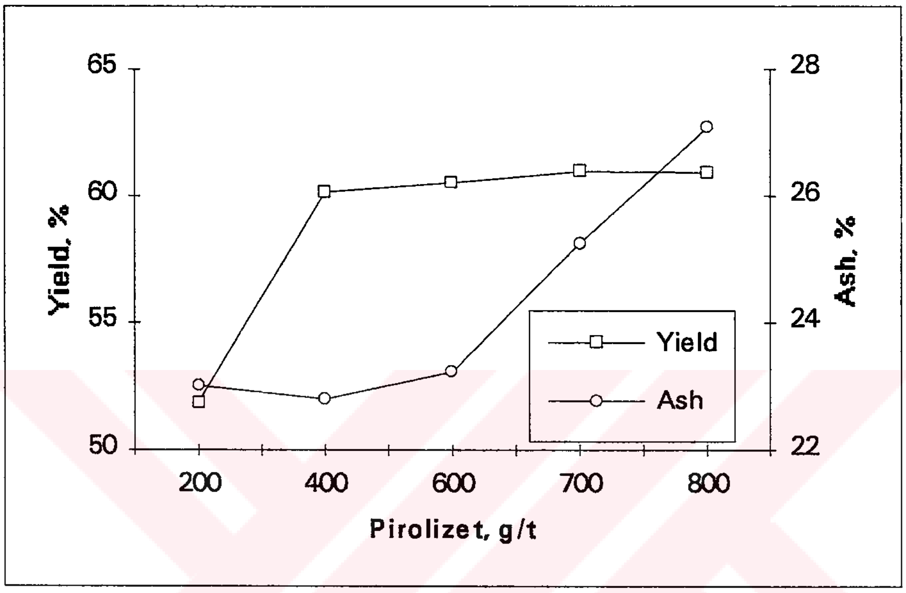

Figure 6. Effect of Pirolizet Amount

As it can be seen from Figure 6, 400 g/t Pirolizet is enough to obtain the clean coal with 60.16 % yield and 22.80 % ash. Ash content increased gradually with further collector consumption, but the yield stayed almost constant. It means that instead of coal some ash particles floated with further addition of Pirolizet. Aplan (1993) stated that excess amount of any reagent is invariably deleterious to the flotation process in terms of the selectivity between the coal and the gangue minerals. It is seen from Figure 4 that very similar concentrates was achieved with 400 g/t Pirolizet and 630 g/t kerosene+80 g/t pine oil or diethyl isohexanol with about 60.0 % yield and 22.5 % ash. But in former case reagent consumption was less than the latter.

4.2.2 Flotation with Ionizing Collectors

4.2.2.1 Flotation with Pamak 1

In the tests, Pamak 1 (a mixture of fatty acids) was used between 900 and 3000 g/t. The results of the tests given in Table 8 are plotted in Figure 7.

The yield and ash content of the concentrate increased with increasing collector consumption. The highest yield is 55.88 % that was achieved with 3000 g/t collector consumption. When the results of Pamak 1 (Figure 7) are compared with the results of kerosene+pine oil (Figure 1), it is seen that very similar concentrates were obtained with 3000 g/t Pamak 1 and 540 g/t kerosene+80 g/t pine oil. The latter concentrate has 22.14 % ash and 57.74 % yield which are only 1 % less and 2 % more than the former, respectively. Figure 7 also showed the possibility of production of very clean coal (e.g., less than 14 % ash) with the use of less than 1200 g/t Pamak 1.

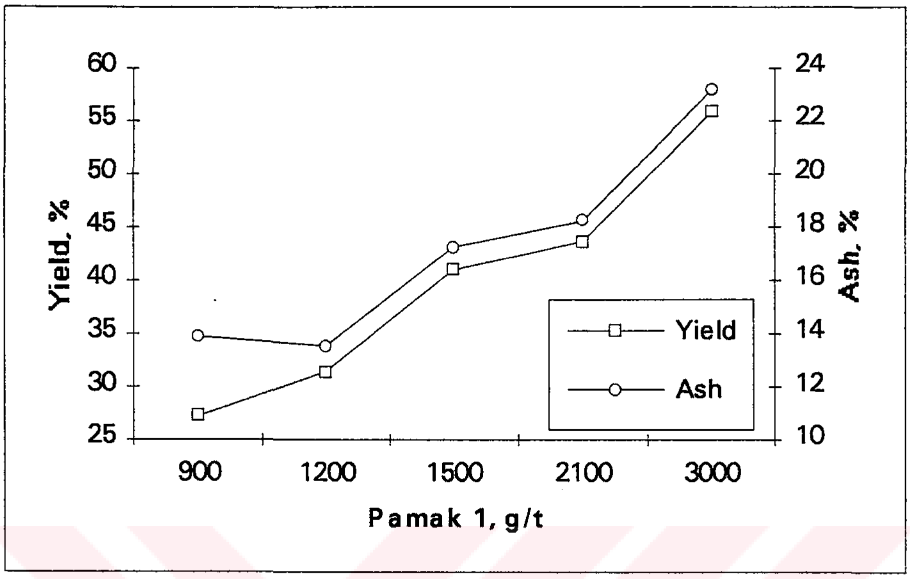

Figure 7. Effect of Pamak 1 Amount

4.2.2.2 Flotation with Pamak 1 at Various pH Values

pH affects the activities of fatty acid species and hence the flotation of minerals (Somasundaran and Sivakumar, 1988). The tests were carried out at pH between 5 and 11 at 1500 g/t Pamak 1 consumption, the results were given in Table 9 and are plotted in Figure 8.

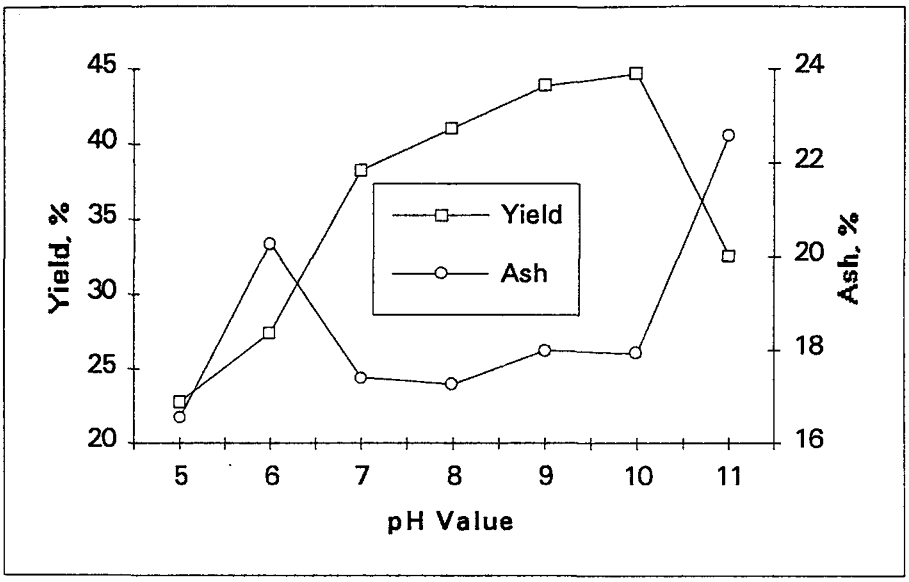

Figure 8. Effect of pH on Pamak 1 Performance

The best results were obtained between pH 8 to 10. It was stated by Crozier (1992) that the long chain fatty acid carboxylates go through an ionization process between pH 4 to 10. At around 8, the ionization is 1:1 ion complex, $RCOOH\text{-}RCOO^-$ and they can act as a dual collector/frother. Somasundaran and Ananthapadmanabhan (1979) concluded that the maximum activity of ionomolecular species ($RCOOH\text{-}RCOO^-$) occurs at pH 7.8, and the activities of total oleate ($RCOO^-$) and the dimer $(RCOO)_2^{-2}$ increase as the pH is increased up to pH 7.8 and then remain constant at higher pH values.

4.2.2.3 Flotation with Pamak 1 and Accoal E12

The effect of addition of Accoal E12 to Pamak 1 was investigated. In these experiments amount of Pamak 1 was kept constant as 1200 g/t, but Accoal E12 was used between 100 and 500 g/t. The results of the tests were given in Table 10 and are plotted in Figure 9.

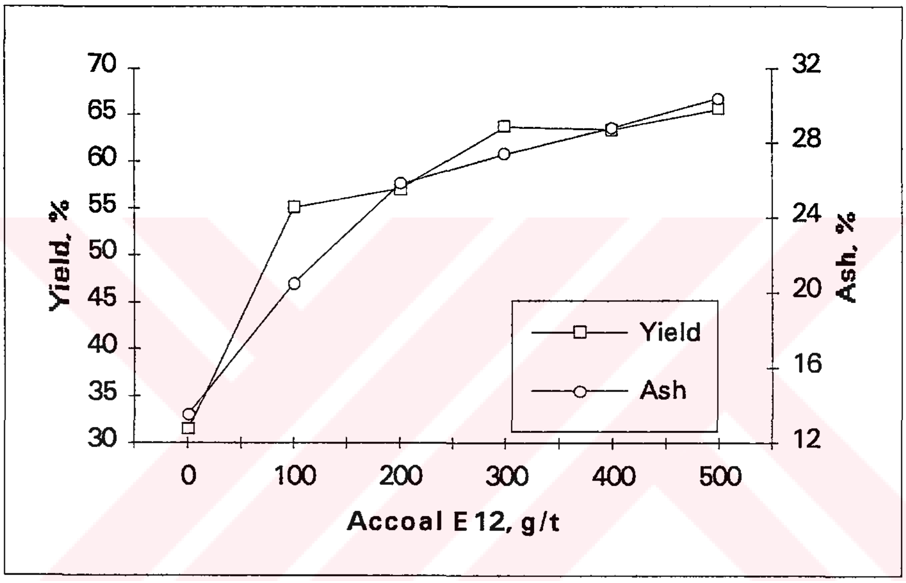

Figure 9. Effect of Accoal E12 Addition

Accoal E12 increased both the yield and ash content of clean coal. But at 100 g/t Accoal E12 consumption, a concentrate with 20.49 % ash and 55.07 % yield was obtained. When Figures 7 and 9 are compared, this concentrate is seen a little better than the 23.20 % ash and 55.88 %

yield concentrate which was achieved with 3000 g/t Pamak 1. It is obvious that the reagent consumption is reduced with promoter Accoal E12, probably due to its emulsification role (Scanlon et al, 1983; and Laskowski, 1986). On the other hand, Accoal E12 addition to kerosene as shown in Figure 1 gave approximately the same results.

4.2.2.4 Flotation with Oleic Acid

Oleic acid was used for the flotation of coal without addition of any frother as done by Hussain (1992). Flotation test results achieved with ionizing oleic acid collector were tabulated in Table 11 and are plotted in Figure 10.

The yield and ash contents of the concentrates increased with the increase in collector consumption. But oleic acid is weaker collector than Pamak 1 (a mixture of fatty acids containing 47.4 % oleic acid in its composition) and it is not as selective as Pamak 1. Myers (1988) stated that more stable emulsions can be obtained with a component than a single surfactant with the same nominal HLB. Hussain (1992) working with Zonguldak coals found Pamak 4 (a mixture of fatty acid containing 46.7 % oleic acid) as a better collector than oleic acid.

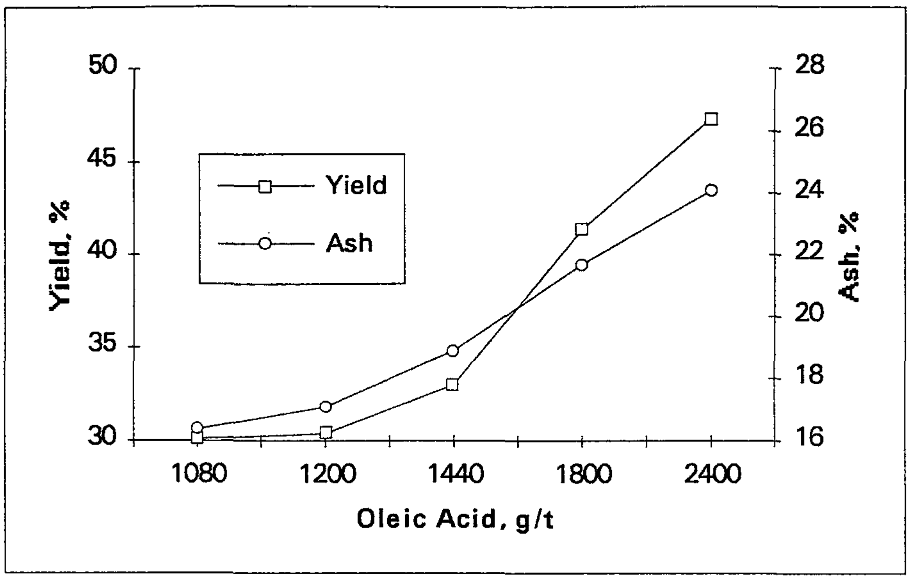

Figure 10. Effect of oleic Acid Amount

4.2.2.5 Flotation with Tall Oil

Tall oil was used between 1040 and 2600 g/t for flotation of coal fines without addition of any frother. The results of the tests given in Table 12 are plotted in Figure 11.

The yield and ash content of concentrates increased with increasing tall oil consumption. Tall oil containing about 40 % oleic acid seems to be stronger collector than oleic acid (Figure 10). Tall oil can be thought as surfactant-alcohol mixture since it contains about 30 % rosin acid which are frothers (Crozier, 1992).

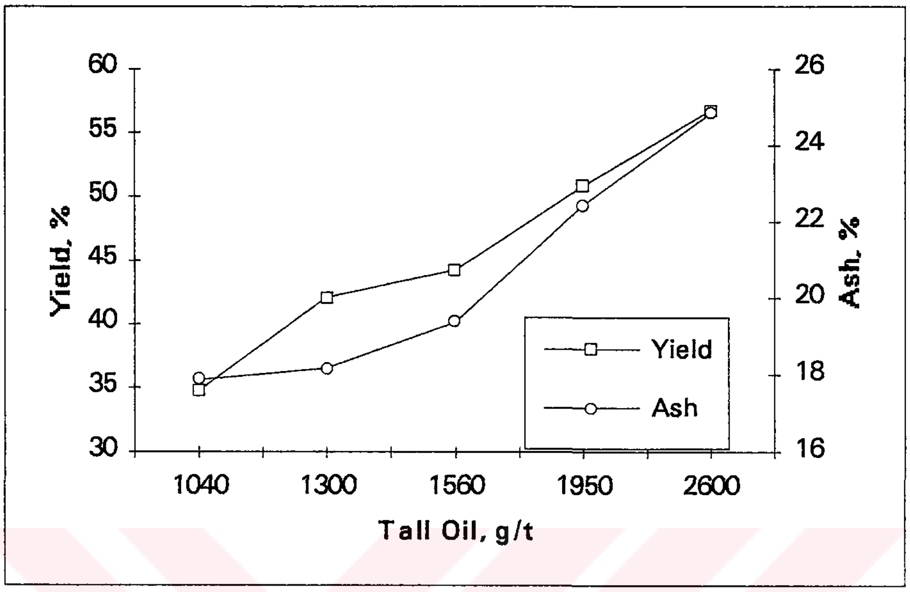

Figure 11. Effect of Tall Oil Amount

4.2.2.6 Flotation with Pamak 4

Pamak 4 was used for flotation of fine coals without addition of any frother as done by Hussain (1992).The results of the tests given in Table 13 are plotted in Figure 12.

The concentrates achieved with Pamak 4 are similar to tall oil and Pamak 1 concentrates, it was not possible to produce high yield (above 60 %).

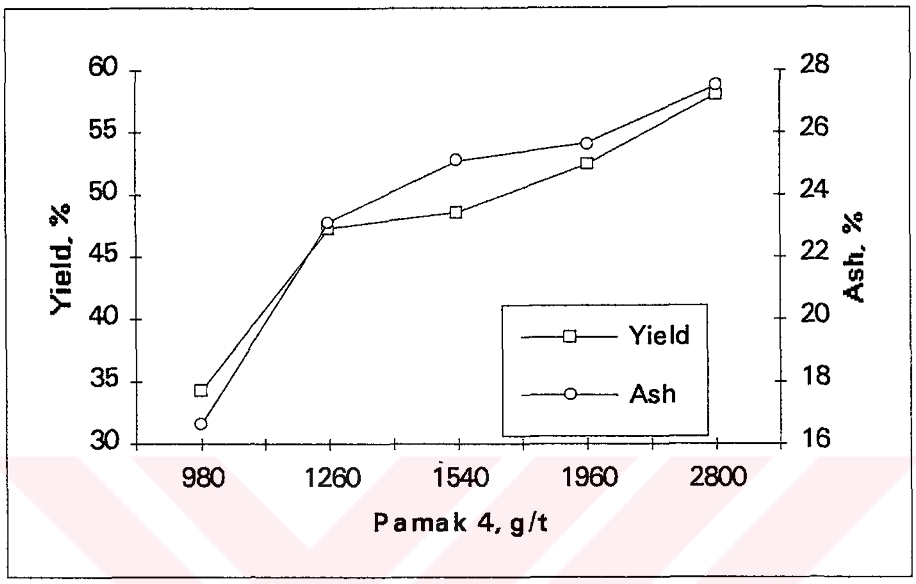

Figure 12. Effect of Pamak 4 Amount

When the results of ionizing collectors are compared with kerosene-frother combination, it was found that all of the ionizing collectors, except Pamak 1, produced coal with low yields. Although Pamak 1 showed comparable results with non-ionizing collectors, its consumption was high as 3000 g/t. On the other hand, the concentrates with low ash content (below 20 %) could only be achieved with ionizing collectors.

4.2.3 Flotation with Sulfonate Type Collectors

Sulfonates are water soluble pastes and have frothing properties. They were fed to the cell as 10 % water solution. The mixtures of sulfonates sometimes give better selectivity in obtaining good grade and recovery without the addition of any auxiliary frothers (Cyanamid, 1986). Aeropromoters 801, 825, 848 and their mixtures with Pamak 1 were used in the tests, because the mixture of fatty acids and sulfonates produces efficient emulsions in flotation as stated by Baarson et al (1962).

4.2.3.1 Flotation with Aeropromoters

Coal flotation with different Aeropromoters was compared at constant collector consumption (1000 g/t). The results were given in Table 14.

As seen from Table 14, Aeropromoters 801 and 848 are very weak collectors when they were used alone. Aeropromoter 825 was relatively better than the others with 36.85 % yield and 19.43 % ash content in the clean coal. American Cyanamid (1986) stated that these promoters tend to produce large volume of froth having little load-carrying capacity, when they were used as principle collectors.

4.2.3.2 Flotation with Aeropromoter 825 at Various pH Values

The effect of pH on the flotation using Aeropromoter 825 was tested at constant amount (1000 g/t) of promoter addition. Experiments were carried out at pH between 4.0 to 8.0 and the results were given in Table 15 and plotted in Figure 13.

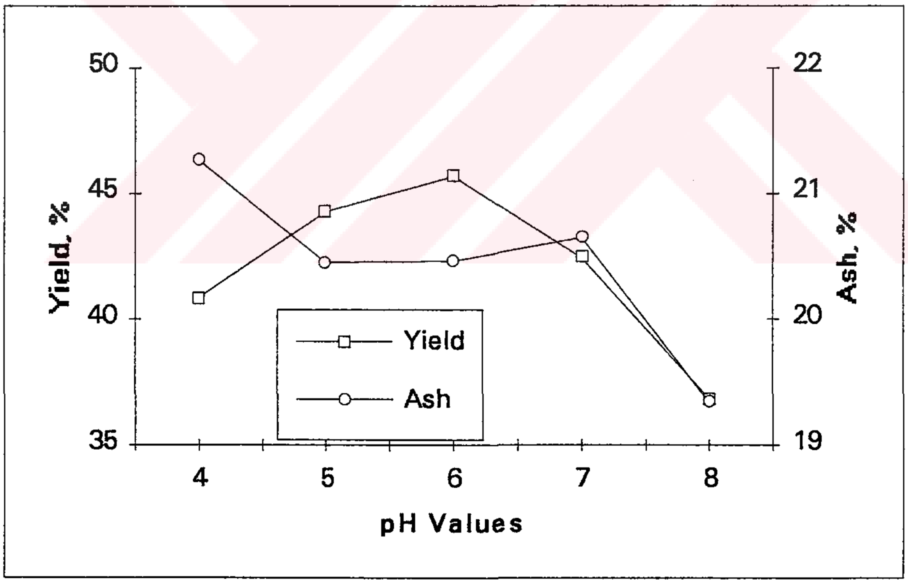

Figure 13. Effect of pH on Aeropromoter 825 Performance

As it is seen from Figure 13, ash content of concentrate did not change much with the change in pH, but there is about 9 % difference in yield between pH 6.0 and 8.0. The best concentrate having 45.68 % yield and 20.46 % ash content in clean coal was obtained with Aeropromoter 825 at pH 6.0.

4.2.4 Flotation with Pamak 1 and Aeropromoter Mixtures

The consumption of Pamak 1 was kept constant as 1200 g/t, but Aeropromoter was used at different amounts between 60 to 240 g/t. in order to determine the best Aeropromoter/Pamak 1 ratio by weight.

4.2.4.1 Flotation with Pamak1 and Aeropromoter 825 Mixtures

The results of the tests, carried out with these mixtures were given in Table 16 and are plotted in Figure 14.

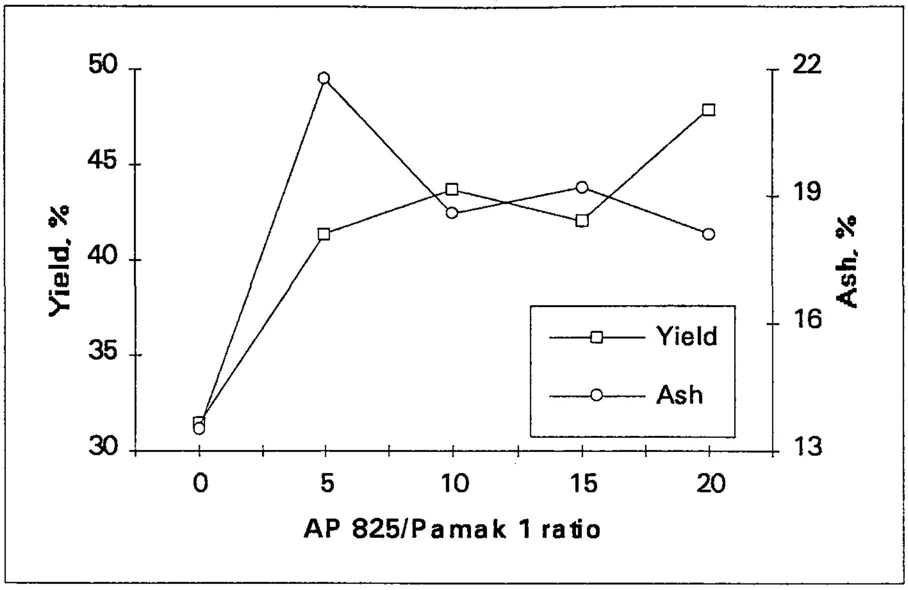

Figure 14. Flotation with Pamak 1 and AP 825 at Different Ratios

It can be seen from Figure 14 that the cleanest concentrate with 18.12 % ash and 47.92 % yield was obtained when the ratio of Aeropromoter 825 to Pamak 1 was 20 % by weight. From comparison of Figures 14 and 7, it may be concluded that this combination reduced collector consumption and increase concentrate yield, since the concentrate having 18.25 % ash, with 43.71 % yield could be obtained with the consumption of 2100 g/t Pamak 1.

4.2.4.2 Flotation with Pamak 1 and Aeropromoter 801 or 848 Mixtures

As can be seen from Tables 17 and 18, no satisfactory results were obtained with the mixture of Aeropromoter-Pamak 1 at any ratio.

CHAPTER V

CONCLUSIONS

1. Zonguldak high ash coal fines can be floated with conventional kerosene-frother combination as well as with a frother alone or with ionizing collectors such as tall oil or Pamak 1. Their effectiveness in flotation vary in terms of concentrate yield and ash content.

2. Kerosene-pine oil combination can be used successfully for the production of concentrates with 52-65 % yield and 21-26 % ash.

3. Among four different frothers, used in combination with kerosene, pine oil and diethyl isohexanol show more selectivity towards coal. Kerosene, when combined with one of these frothers, causes the production of a clean coal having about 22 % ash with about 60 % yield. The consumption of non-polar oil and frothers are 630 and 60 g/t, respectively.

4. The use of pine oil alone for the flotation of Zonguldak high ash coal fines shows the possibility of production of concentrates with less than 20 %

ash. The addition of an oily collector, kerosene, increased coal floatability and hence the concentrate yield.

5. It is possible to produce a concentrate containing 22.80 % ash with 60.16 % yield by using only 400 g/t Pirolizet, a Polish coal collector+frother combination.

6. Carboxylate type ionizing collectors produce more cleaner concentrates than conventional collectors. Pamak 1 with the consumption lower than 1200 g/t can produce clean coals containing less than 14 % ash. On the other hand, the cleanest coal obtained with conventional reagent contains 19.28 % ash which was achieved with 160 g/t pine oil. The common disadvantage of all ionizing collectors is their low yield.

7. No appreciable difference is found between the results of sulfonate and carboxylate collectors. Premixing sulfonate-carboxylate collectors reduces collector consumption reasonably, but cannot increase concentrate yield very much.

8. Although ionizing collectors are more active and selective toward high ash coal fines, due to low concentrate yield, their economic judgement must be done before industrial application.

9. Fatty acid mixture collectors containing more than 40 % oleic acid, e.g., Pamak 1, Pamak 4 and Tall oil are found better collectors than oleic acid alone.

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ASTM D 3174-73 Ash in the Analysis Sample of Coal and Coke.

ASTM D 3175-89a Volatile Matter in The Analysis Sample of Coal and Coke.

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APPENDIX

APPENDIX

TABLES

Table 1. Proximate Analysis of Dried Coal Sample

Ash, %	Volatile Matter, %	Fixed Carbon, %
46.10	19.27	34.63

Table 2. Effect of Kerosene at Constant Pine Oil Consumption

Kerosene Addition		Clean Coal	
g/t	drops	Yield, %	Ash, %
360	4	52.67	21.95
540	6	57.74	22.14
810	9	62.77	24.90
1080	12	63.12	26.35
1350	15	65.09	26.40

Table 3. Effect of Pine oil at Constant Kerosene Consumption

Pine Oil Addition		Clean Coal	
g/t	drops	Yield, %	Ash, %
40	1	56.52	27.27
80	2	57.74	22.14
120	3	64.54	28.38
160	4	62.22	27.73

Table 4. Effect of Accoal E12 Amount

Accoal E12 Addition		Clean Coal	
g/t	drops	Yield, %	Ash, %
100	1	55.52	20.21
200	2	59.39	22.30
300	3	62.46	25.54
400	4	64.38	25.60
500	5	64.35	27.74

Table 5. Comparison of Different Frothers

Frother Type	Clean Coal	
	Yield, %	Ash, %
Pine Oil	59.58	22.62
Diethyl Isohexanol	60.03	22.67
Dowfroth 250	63.34	25.62
Methyl Isobutyl Carbinol	53.87	23.33

Table 6. Effect of Pine Oil Amount

Pine Oil Addition		Clean Coal	
g/t	drops	Yield, %	Ash, %
40	1	37.22	25.09
80	2	43.34	19.63
160	4	48.17	19.28
240	6	51.51	21.45
320	8	52.56	22.29

Table 7. Effect of Pirolizet Amount

Pirolizet Addition		Clean Coal	
g/t	drops	Yield, %	Ash, %
200	2	51.89	23.01
400	4	60.16	22.80
600	6	60.52	23.23
700	7	60.98	25.24
800	8	60.92	27.08

Table 8 Effect of Pamak 1 Amount

Pamak 1 Addition		Clean Coal	
g/t	drops	Yield, %	Ash, %
900	6	27.24	13.89
1200	8	31.46	13.53
1500	10	41.03	17.27
2100	14	43.71	18.25
3000	20	55.88	23.20

Table 9. Effect of pH on Pamak 1 Performance

pH	5	6	7	8	9	10	11
Clean Coal Yield, %	22.83	27.41	38.21	41.03	43.82	44.67	32.56
Clean Coal Ash, %	16.56	20.28	17.42	17.27	17.99	17.93	22.57

.Table 10. Effect of Accoal E12 Amount

Accoal E12 Addition		Clean Coal	
g/t	drops	Yield, %	Ash, %
0	0	31.46	13.53
100	1	55.07	20.49
200	2	57.10	25.79
300	3	63.68	27.38
400	4	63.39	28.81
500	5	65.60	30.39

Table 11. Effect of Oleic Acid Amount

Oleic Acid Addition		Clean Coal	
(g/t)	drops	Yield, %	Ash, %
1080	9	30.11	16.38
1200	10	30.40	17.09
1440	12	33.01	18.90
1800	15	41.33	21.69
2400	20	47.33	24.07

Table 12. Effect of Tall Oil Amount

Tall Oil Addition		Clean Coal	
g/t	drops	Yield, %	Ash, %
1040	8	34.78	17.88
1300	10	42.09	18.17
1560	12	44.24	19.38
1950	15	50.81	22.43
2600	20	56.76	24.84

Table 13. Effect of Pamak 4 Amount

Pamak 4 Addition		Clean Coal	
g/t	drops	Yield, %	Ash, %
980	7	34.35	16.66
1260	9	47.26	23.12
1540	11	48.56	25.09
1960	14	52.41	25.65
2800	20	58.0	27.49

Table 14. Comparison of Three Aeropromoters

Aeropromoter	Clean Coal	
	Yield, %	Ash, %
Aeropromoter 801	12.23	34.07
Aeropromoter 825	36.85	19.34
Aeropromoter 848	13.15	33.76

Table 15. Effect of pH on AP 825 Performance

pH Values	4	5	6	7	8
Clean Coal Yield, %	40.87	44.29	45.68	42.45	36.85
Clean Coal Ash, %	21.26	20.45	20.46	20.65	19.34

Table 16. Effect of Pamak 1 and Aeropromoter Mixtures

AP 825/Pamak 1 ratio, %	AP 825 g/t	Clean Coal Yield, %	Clean Coal Ash, %
0	0	31.46	13.53
5	60	41.38	21.78
10	120	43.70	18.59
15	180	42.03	19.23
20	240	47.92	18.12

Table 17. Effect of Pamak 1 and Aeropromoter 801 Mixtures

AP 801/Pamak 1 ratio, %	AP 801 (g/t)	Clean Coal	
		Yield, %	Ash, %
0	0	31.46	13.53
5	60	41.23	20.93
10	120	40.24	21.14
15	180	38.56	20.72
20	240	38.34	20.45

Table 18. Effect of Pamak 1 and Aeropromoter 848 Mixtures

AP 848/ Pamak 1 ratio, %	AP 848 (g/t)	Clean Coal	
		Yield, %	Ash, %
0	0	31.46	13.53
5	60	36.29	18.92
10	120	44.85	21.62
15	180	37.60	20.21
20	240	38.24	24.46

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