

CLEANING OF ŞIRNAK KARATEPE ASPHALTITES

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ABSTRACT

CLEANING OF ŞIRNAK KARATEPE ASPHALTITES

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Asphaltite is petroleum originated substance and formed by metamorphism. Turkey has 82 million tons of asphaltite reserves in Şırnak and Silopi region of Southeastern part of Anatolia. The proximate analysis of Şırnak Karatepe asphaltite sample indicated that the ash and sulfur content were 46.86 % and 5.56 %, respectively.

In this study, Şırnak Karatepe asphaltite sample was concentrated by gravity separation and flotation methods. The aim of this research was to decrease the ash and sulfur content below 20% and 2%, respectively, which are the requirements of Şırnak Municipality.

Gravity based cleaning equipments such as multi gravity separator, shaking table and Falcon concentrator gave no satisfactory results in terms of ash and sulfur removal. The products with lowest ash content were obtained with Falcon concentrator after two stage cleaning of -100 micrometer feed.

Flotation parameters of Karatepe asphaltite were also examined during the study. As a result of multi-stage flotation with stage addition of Collector Accoal 9630 and depressant Na_2SiO_3 , ash content of asphaltite was decreased to 17.59 % with 15.31 % combustible recovery. Even though the ash content specification met by flotation, the sulfur content of cleaned asphaltite (6.68 % S) was more than the sulfur limit of Şırnak Municipality.

Keywords: Asphaltite, Gravity Separation, flotation

ÖZ

ŞIRNAK KARATEPE ASFALTİTLERİNİN TEMİZLENMESİ

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Asfaltit, petrol kökenli bir maddedir ve metamorfoz sonucu oluşmuştur. Türkiye'nin Güneydoğu Anadolu Bölgesinde Şırnak ve Silopi bölgelerinde 82 milyon ton asfaltit rezervi vardır. Şırnak Karatepe asfaltit numunesinin kısa analiz sonuçları, kül ve kükürt içeriğinin sırasıyla % 46.86 ve % 5.56 olduğunu göstermiştir.

Bu çalışmada Şırnak Karatepe asfaltit numunesinin gravite ayırımı ve flotasyon yöntemleriyle zenginleştirilmesine çalışılmıştır. Çalışmanın amacı, asfaltitin kül ve kükürt içeriğini Şırnak Belediyesi'nin konut ısınması için kullanılacak katı yakıtlar için sınır olarak belirlemiş olduğu, sırasıyla %20 ve %2 değerlerinin altına düşürmektir.

Multi gravite separatör, sallantılı masa ve Falcon konsantratörü gibi gravite tabanlı temizleme ekipmanları kül ve kükürt giderimi için tatmin edici bir sonuç

vermemiştir. En düşük kül içeriđi -100 mikron boyutundaki numunenin Falcon konsantratöründe 2 kere temizlenmesiyle elde edilmiştir.

Çalıřma süresince Karatepe asfaltinin flotasyon parametreleri de incelenmiştir. Kademeli olarak Accoal 9630 kollektörü ve Na_2SiO_3 bastırıcısı ilavesiyle yürütölen çok temizlemeli flotasyon çalışmasıyla asfaltin kül içeriđi %15.31 yanabilir verimiyle %17.59'a düşürölmüştür. Her ne kadar kül içeriđi flotasyonla sağlanmışsa da temiz asfaltin kükürt içeriđi %6.68'lik değeriyle řırnak Belediyesi kükürt sınırının üstünde kalmıştır.

Anahtar Kelimeler: Asfaltit, Gravite konsantrasyonu, flotasyon

to my wife Senem Demir

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CHAPTER 1

INTRODUCTION

1.1 General Remarks

By increasing population and effect of consumption culture need of energy increased dramatically and this increase is going to continue. World energy demand was 4672 MTEP (million tons petroleum equivalent) in 1973, 8084 MTEP in 2006 and foreseen to be 16000 MTEP in 2030 (IEA, 2008). In recent years, need of energy became the major interest area in the world. Diminishing oil reserves are enforcing people to find and use new energy sources.

Like other countries, energy demand and consumption of Turkey is increasing as well. The electricity energy demand of Turkey in 1990 was 56,811.7 GWh and in 2007 was 190,000 GWh. Table 1.1 shows the annual development of Turkey's gross electricity generation and demand (TEİAŞ, 2008). As can be seen from Table 1.1 Turkey can hardly meet the electricity energy demand. The required electrical energy of Turkey is primarily met from thermal and hydraulic sources. Turkey is an energy importing country. 81.4 % of energy used in Turkey in 2007 is imported (ETKB, 2008).

Contrary to this situation, asphaltite deposits in the Southeastern Anatolia Region of Turkey, which is roughly 82 million tons with net calorific value of 2876–5536 kcal/kg, are idle. The production quantities are given in Table 1.2 (DPT, 2007). This production is mainly used in domestic heating. Table 1.3 shows the distribution of asphaltite reserves of Turkey.

Table 1.1 Annual development of Turkey's gross electricity generation and demand (TEİAŞ, 2008)

NATIONAL GENERATION (GWh)						GROSS DEMAND	
YEARS	THERMAL	HYDRO	GEOTHERMAL+ WIND	TOTAL	INCREASE %	GWh	INCREASE %
1990	34,315.3	23,147.6	80.1	57,543.0	10.6	56,811.7	8.0
1995	50,620.5	35,540.9	86.0	86,247.4	10.1	85,551.5	10.0
2000	93,934.2	30,878.5	108.9	124,921.6	7.3	128,275.6	8.3
2005	122,242.3	39,560.5	153.4	161,956.2	7.5	160,794.0	7.2
2006	131,835.1	44,244.2	220.5	176,299.8	8.9	174,637.3	8.6
2007	155,196.2	35,850.8	511.1	191,558.1	8.7	190,000.2	8.8

Table 1.2 Production Quantities of asphaltite in Turkey Between 1980 -2005 (DPT, 2007)

Production quantities of asphaltite in Turkey between 1980–2005 (1000 tons)											
Year	1980	1985	1990	1992	1994	1996	1998	2000	2002	2004	2005
Quantity	558	523	539	475	117	109	93	107	280	728	668

Table 1.3 Asphaltite reserves of Turkey (DPT, 2007)

Area	Reserve (1000 tons)			
	Proven	Probable	Possible	Total
Silopi Harbul	17 914	7 851	-	25 765
Silopi Silip	3 071	1 335	-	4 406
Silopi Üçkardesler	9 472	10 881	-	20 353
Şırnak Avgamasya	7 481	673	-	8 154
Şırnak Milli	2 000	2 900	1 600	6 500
Şırnak Karatepe	500	2 000	2 500	5 000
Şırnak Seridahli	3 534	1 254	1 279	6 067
Şırnak Nivekara	300	1 000	700	2 000
Şırnak Ispindoruk	100	500	500	1 100
Şırnak Segürük	550	450	-	1 000
Şırnak Rutkekurat	-	-	1 000	1 000
Şırnak Uludere Ortasu	551	53	-	604
Total	45 473	28 897	7 579	81 949

1.2 Asphaltic Substances and Asphaltites in Southeastern Turkey

It is known that the asphaltic substances are formed as a result of the petroleum substances change called *metamorphism*. In this change, effects like time, heat and pressure had a role. Depending to the gradual stages of progress of the metamorphosis, material types with different characteristics were formed.

1.2.1 Transformation of Asphaltic Substances

Figure 1.1 (Orhun, 1969) shows the formed asphaltic substances with different characteristics with respect to the extent of metamorphosis which the petroleum was exposed to:

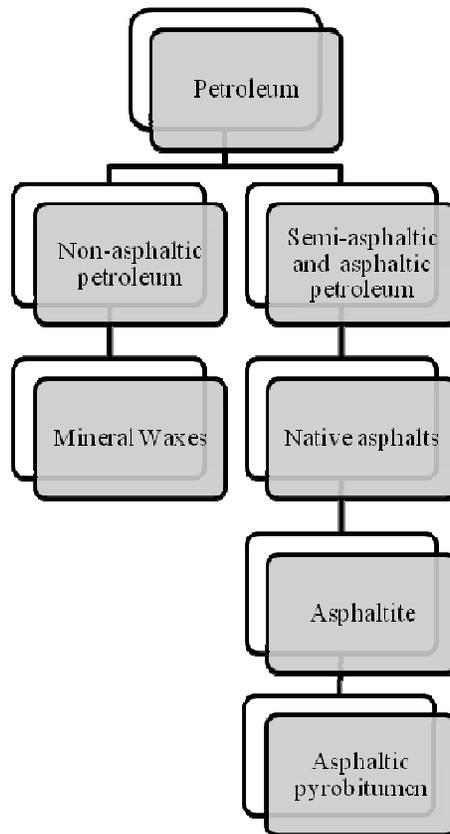


Figure 1.1 Transformation of asphaltic substances

As seen from Figure 1.1 the petroleum passed from various gradual stages: first soft natural asphalts, then harder asphalts and finally to asphaltic pyrobitumens. Asphaltic pyrobitumens stands for the last stage of metamorphosis.

The following are the most important characteristic properties of these materials:

Natural asphalts: Dark colored, with various hardness, relatively less volatile, essentially composed of hydrocarbons, contain very little or none of any oxygenated compounds or pyrocrystalline paraffins, melt when heated, can largely dissolve in carbon sulfur and can give sulfonation products.

Asphaltites: Dark colored, relatively hard and in volatile solid substances. Essentially composed of hydrocarbons, contain very little or none of any oxygenated compounds or pyrocrystalline paraffins, melts difficultly when heated, melting points are high (about 120–315°C), dissolve in carbon sulfur; sulfonation products don't dissolve in water.

Asphaltic pyrobitumens: Dark colored, relatively hard and in volatile solid substances. Essentially composed of hydrocarbons, contain very little or none of any oxygenated compounds, don't melt when heated and don't dissolve in carbon sulfur.

According to Nellenstein asphaltic pyrobitumens contain three main groups (Nellenstein, 1938):

1. Medium (petroleum constituents)
2. *Lyophilic* part or protective part (corresponds to asphaltic resins)
3. *Lyophobic* part (composed of colloidal particles of elementary carbon)

The protective part which corresponds to asphaltic resins is generally composed of high, unsaturated hydrocarbons. There is an adsorption relation between the components of second and third parts; these form a disperse phase and asphalt micelle, correspond to asphaltens.

According to this theory, asphalt components can be classified as the following system:

Petrolens (maltenes) and petroleum compounds: petroleum medium

Asphalt acids + asphalt acid anhydrites + asphaltic resins: small amount of carbon and large amount of protective part

Asphaltenes: carbon and important amount of protective part

Carbenes: carbon and little amount of protective part

Carboids or pyrobitumens: carbon and very little amount of protective part

Asphaltenes, resins and petroleum constituents form a colloidal system together. In this system asphaltenes are dispersed in petroleum constituents and the solution is stabilized with the help of resin fraction which acts as protective colloid.

Asphaltic bitumen is a largely protected *lyophobic sol* and each asphaltene micelle inside it has a carbon core surrounded by hydrocarbon layers. Properties of asphaltic materials depend on the concentration of disperse phase, dispersion degree and properties of the medium. The combination of these functions causes formation of various materials such as soft, viscous asphalts and asphaltites. There is a little amount of asphalt micelles in petroleum and forms a disperse solution. During the metamorphosis of petroleum, the concentration of petroleum decreases due to evaporation, corresponding to that the particle sizes of asphalt micelles increase and the products stand for a disperse system varying from soft to medium hard asphalt. During metamorphosis a lot of chemical reactions such as oxidation, polymerization and condensation occur and hydrocarbon molecules transform to higher complexity molecules which have higher molecular weights. Even though natural asphalts are evaporation products of petroleum, asphalts are products of reaction and transformation rather than evaporation. The polymerization of molecules by condensation is accepted as the main factor of formation of petroleum constituents and resins. Asphaltenes transform into carbenes by polymerization and these transform into carboids by condensation. Petrolenes, asphaltenes and carbenes are fractions of asphaltic bitumens which dissolve in carbon sulfur; carboids do not dissolve in carbon sulfur.

The characteristic properties of asphaltic material types in the world are given in Table 1.4 (Abraham, 1960). As seen on Table 1.4, asphaltites are characterized by high melting points (about 120–315°C), natural asphalts are softened about 15–160°C. Mohs ruler hardness is 1 or below for natural asphalts and varies between 2 and 3 for asphaltites.

Most important distinctive characteristics of asphaltites and asphaltic pyrobitumens are melting by heat effect and solubility in carbon sulfur. Asphaltites can melt by heat effect and dissolve in carbon sulfur (Gilsonite 99–100%, *glance* tar 97–100% and grahamite —in base without mineral substance— 90–100%). Asphaltic pyrobitumens which represent the last stage of metamorphosis don't melt when heated and dissolve very little in carbon sulfur (wurtzilite 5–10%, albertite 2–10%, impsonite trace quantity–6%). Because of this, it is concluded that the solubility in carbon sulfur is related to the degree of metamorphosis in asphaltic materials which are at the gradual transition stages from asphaltite to asphaltic pyrobitumen.

1.2.2 Classification of Asphaltic Occurrences in Southeastern Turkey

The values of solubility in carbon sulfur of occurrences in Southeastern Turkey and asphaltic material types in the world (in base without water and mineral) are shown in Figure 1.2 comparatively. According to H. Abraham, wurtzilite, albertite and impsonite whose solubility in carbon sulfur does not exceed 10% are classified as asphaltic pyrobitumen (Table 1.4). Materials with high solubilities in carbon sulfur (in base without minerals) such as Gilsonite (99–100%), *glance* tar (97–100%) and grahamite (90–100%) are classified into asphaltite class.

Table 1.3 The most important distinguishable characteristics of asphaltic materials (Abraham, 1960)

Characteristic features	Petroleums			Natural asphalts (mineral matter less than 10%)	Asphaltites			Asphaltic pyrobitumens		
	Non-asphaltic	Semi-asphaltic	Asphaltic		Gilsonite	Glance Pitch	Grahamite	Wurtzite	Albertite	Impsonite
Mass color	Brown - Black	Brown - Black	Black	Black	Black	Black	Black	Black	Brown - Black	Black
Failure of rupture	-	-	-	Cochooidal-incised	Cochooidal-incised	Cochooidal-incised	Cochooidal-incised	Cochooidal	Cochooidal-incised	Incised
Luster	-	-	-	Very shiny-dull	Very shiny-a bit shiny	Shiny-dull	Shiny-dull	Shiny	Shiny-dull	Semi-dull
Streak	-	-	-	Dark brown-black	Brown	Brown-black	Black	Light brown	Brown-black	Black
Specific Gravity	0.75-0.90	0.80-0.95	0.85-1.00	0.95-1.12	1.03-1.10	1.10-1.15	1.15-1.20	1.05-1.07	1.07-1.10	1.10-1.25
Hardness (from Mohs Scale)	-	-	-	<1-1	2	2	2-3	2-3	2	2-3
Penetration, @ 5 ^o C	Liquid	Liquid	Liquid	0-350	0-3	0-5	0	0-5	0	0
Melting point, ^o C	<0	<0	<0	15-160	120-175	120-175	175-315	Not melt	Not melt	Not melt
Fixed carbon, % in base without water and mineral	0.5-2	2.5	5-10	1-25	10-20	20-35	35-55	5-25	25-50	50-85
Oxygen, % (without minerals, at essential part)	0-2	0-3	0-5	0-2	0-2	0-2	0-2	0-2	0-3	0-3
Minerals, %	0-1	0-1	0-1	0-10						
Solubility in CS ₂ , %	98-100	98-100	98-100	90-100	98-100	95-100	45-100	5-10	2-10	
Solubility in CS ₂ , % (without minerals, at essential part)	98-100	98-100	98-100	94-100	99-100	97-100	90-100	5-10	2-10	
Non-mineral substance Not dissolved in CS ₂ , % (non-mineral base)	0-0.5	0-1	0-1	0-6	0-1	0-3	0-5	85-95	85-98	93-99
Non-mineral substance Not dissolved in CS ₂ , %	0-0.5	0-1	0-1	0-6	0-1	0-3	0-10	90-95	90-98	94-100
Solubility in petroleum naphtha	98-100	95-100	90-100	10-90	10-60	15-50				
Carbenes %	0-0.5	0-1	0-1	0-5	0-0.5	0-1	0-80			
Sulfonation residue %	90-100	85-95	80-95	90-100	85-95	85-95	80-95	90-98	90-98	90-98
Diazo reaction	Negative	Negative	Negative	Negative	Negative	Negative	Negative	Negative	Negative	Negative
Anthraquinone reaction	Negative	Negative	Negative	Negative	Negative	Negative	Negative	Negative	Negative	Negative

As seen on Figure 1.2 some occurrences in Southeastern Turkey are in the form of asphaltic pyrobitumens; some, although bearing several common characteristics with the pyrobitumens, from the point of metamorphosis, have not progressed as far as the asphaltic pyrobitumen. Still some other substances have characteristics between asphaltites and asphaltic pyrobitumens.

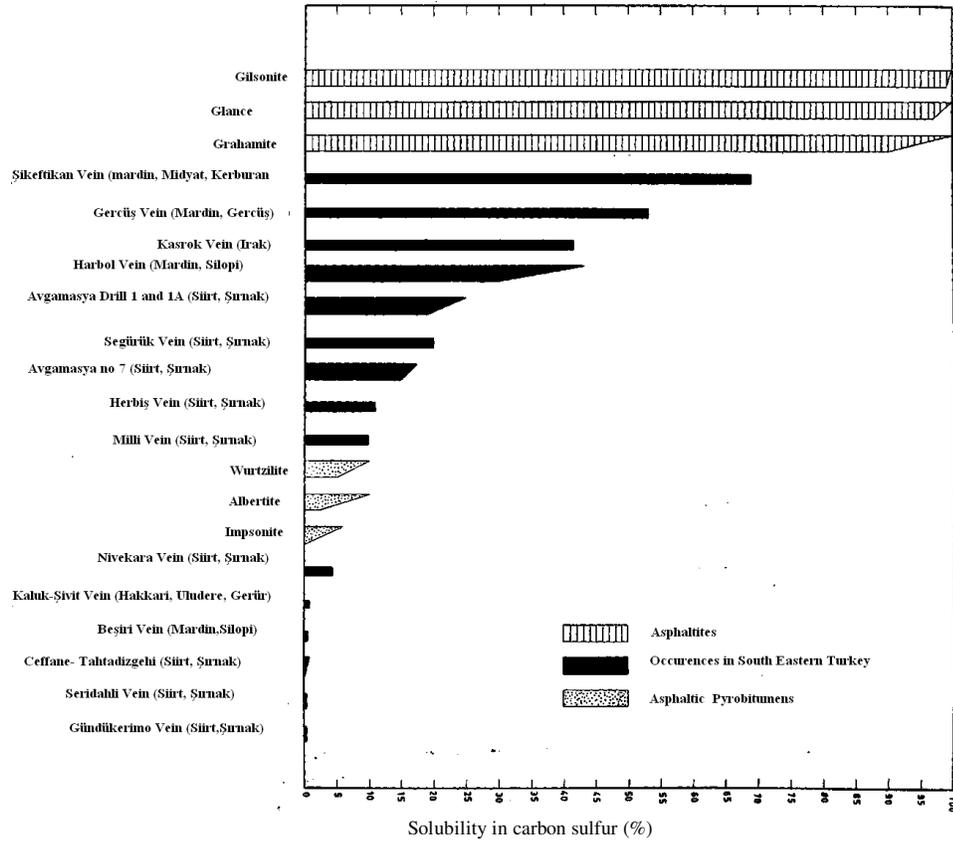


Figure 1.2 Comparisons of the values of solubility in carbon sulfur of occurrences in Southeastern Turkey and the world (Orhun, 1969).

Orhun proposes classification system shown in Table 1.4 for the occurrences in Southeastern Turkey (Orhun, 1969)

Table 1.4 Classification for occurrences in Southeastern Turkey (Orhun, 1969)

Solubility in CS ₂ , %	Species
0–15	Asphaltic pyrobitumen
15–25	Substance close to asphaltic pyrobitumen
25–90	Substance between asphaltite and asphaltic pyrobitumen

1.3 Objective of the Thesis

The objective of this research is to find an asphaltite cleaning process to decrease ash and sulfur content to 20% and 2%, respectively, from Şırnak Karatepe asphaltites.

CHAPTER 2

LITERATURE REVIEW

2.1 Asphaltite Cleaning Methods

Gravity separation and flotation are the possible asphaltite cleaning methods. In the following sections basic procedures of some gravity based equipments and flotation are summarized.

2.1.1 Multi-Gravity Separator

The principle of the Mozley *Multi-Gravity Separator* (MGS) can be visualised as rolling the horizontal surface of a conventional shaking table into a drum, then rotating it so that many times the normal gravitational pull can be exerted on the mineral particles as they flow in the water layer across the surface. Figure 2.1 shows a cross-section of the pilot scale MGS. The Industry Scale MGS consists of two slightly tapered open-ended drums, mounted "back to back", rotating at speeds variable between 90 and 150 rpm, enabling forces of between 5 and 15 G to be generated at the drum surfaces. A sinusoidal shake with an amplitude variable between 4 and 6 cps is superimposed on the motion of the drum, the shake imparted to one drum being balanced by the shake imparted to the other, thus balancing the whole machine. A scraper assembly is mounted within each drum on a separate concentric shaft, driven slightly faster than the drum but in the same direction. This scrapes the settled solids up the slope of the drum, during which time they are subjected to counter-current washing before being discharged as concentrate at the open, outer, narrow end of the drum. The lower density minerals,

along with the majority of the wash water, flow downstream to discharge as tailings via slots at the inner end of each drum (Wills, 2006).

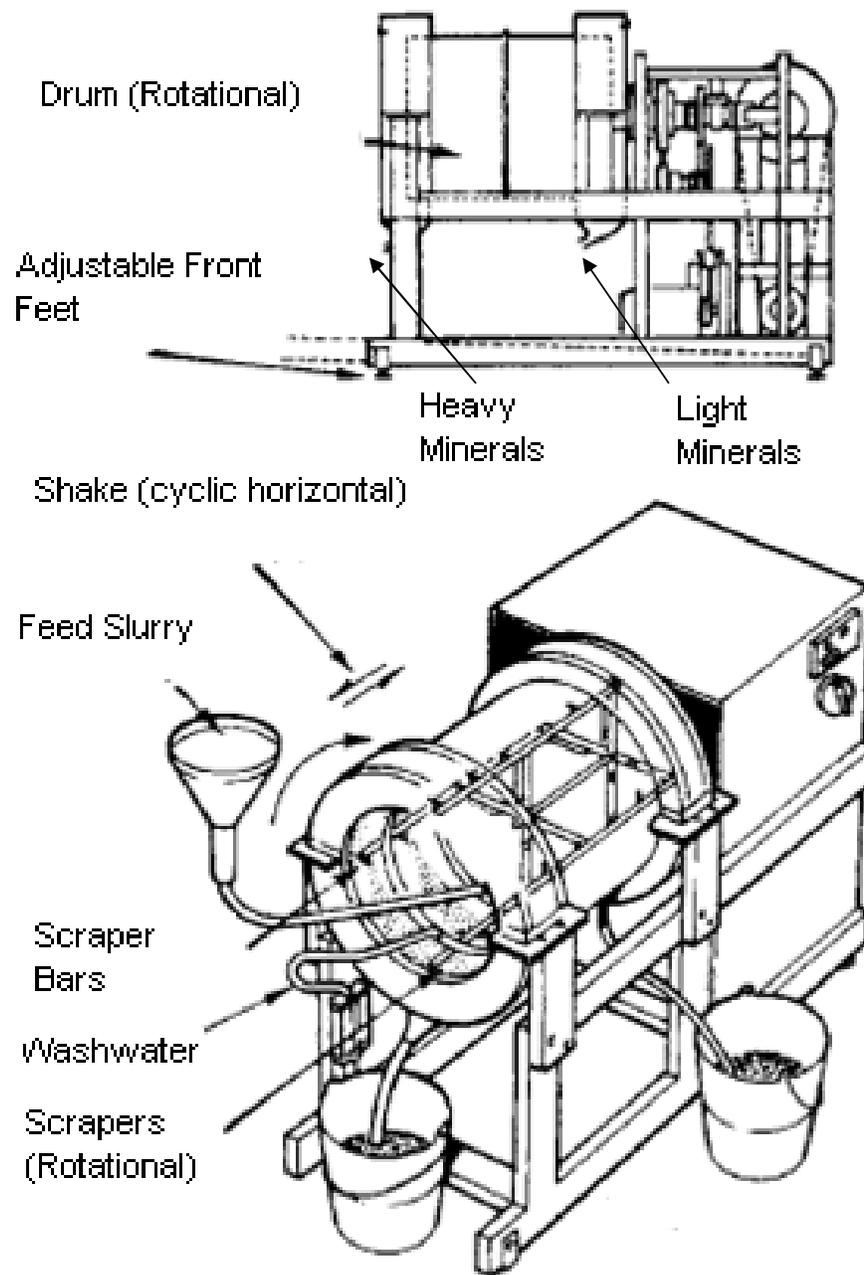


Figure 2.1 Pilot scale MGS

2.1.2 Falcon Concentrator

The Falcon centrifugal concentrator is an enhanced gravity separator which utilizes a large centrifugal force to cause deposition and stratification of the fine particles against the inside of a smooth centrifugal bowl. Figure 2.2 shows a schematic illustration of the separation that occurs inside the Falcon concentrator (Honaker, 1996). The feed is introduced at the bottom of the bowl and onto a spinning rotor. An impeller hurdles the feed against the wall of the rotor. The bottom of the rotor is called the migration zone, and is inclined at a slight angle so that the enhanced gravity field generated by the spinning rotor can be resolved into two force components. The strong component normal to the wall is the concentrating gravity field that provides the strong g forces for the hindered settling processes and stratifies the feed. The weak driving component parallel to the inclined rotor surface pushes the stratified solids up toward the top of the rotor.

The angle of incline on the rotor surface changes near the top of the rotor so that it is now parallel to the axis of rotation. As a result, there is no weak gravity force component to drive particles upward toward the top of the rotor. This part of the rotor is called the retention zone. Light particles on the outside of the bed move upward to the overflow lip of the rotor using the momentum they accumulated in the migration zone and the force of the upward flowing water film. Heavy particles near the bowl surface have neither the speed nor the aid of the water film to cross the retention zone and, thus, become beached on the surface of the rotor. The thickness of the heavy particle layer increases until particles have built up enough into the stream of the flowing water to be carried over the lip. In a batch concentrator, the building of a maximum accumulation of heavy particles against the wall of the rotor in the retention zone signals the time to stop the concentrating process and wash the heavy particles out of the retention zone. In the continuous unit, a slot in the retention zone is connected to withdraw the heavy particles. (Honaker, 1994)

Table 2.1 is showing the corresponding G values to frequency for Falcon concentrator.

Table 2.1 Corresponding G values for Frequency of Falcon concentrator

Frequency (Hz)	Acceleration (G)
20	20
25	31
30	44
35	60
40	78
45	99
50	123
55	148
60	176
68	225
71	250
75	275
78	300

2.1.3 Shaking Table

When a flowing film of water flows over a flat, inclined surface the water closest to the surface is retarded by the friction of the water absorbed on the surface; the velocity increases towards the water surface. If mineral particles are introduced into the film, small particles will not move as rapidly as large particles, since they will be submerged in the slower-moving portion of the film. Particles of high specific gravity will move more slowly than lighter particles, and so a lateral displacement of the material will be produced (Figure 2.3).

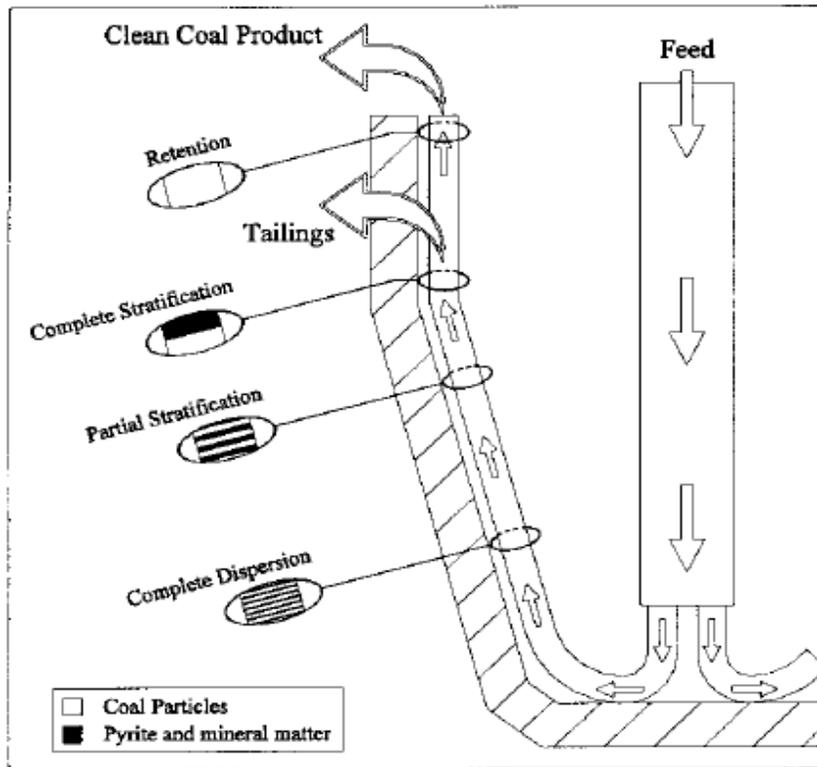


Figure 2.2 A schematic illustration of the operating principles of the continuous Falcon concentrator

The flowing film effectively separates coarse light particles from small dense particles, and this mechanism is utilized to some extent in the shaking table concentrator (Figure 2.4) which is perhaps the most metallurgically efficient form of gravity concentrator, being used to treat the smaller, more difficult flow-streams, and to produce finished concentrates from the products of other forms of gravity system.

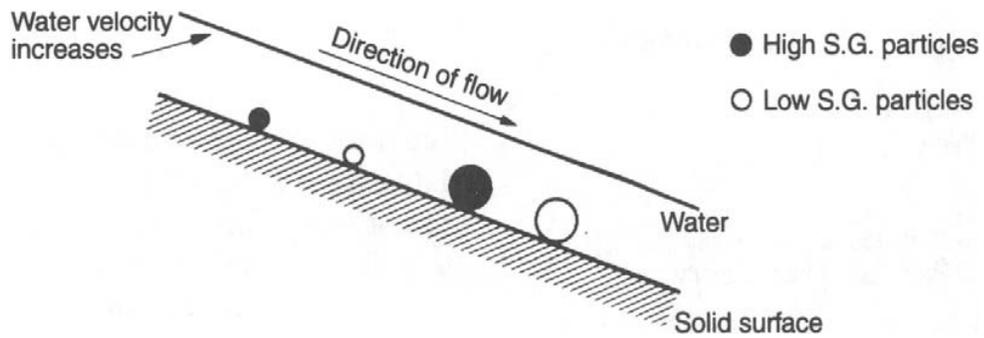


Figure 2.3 Action in a flowing film (Wills, 1985)

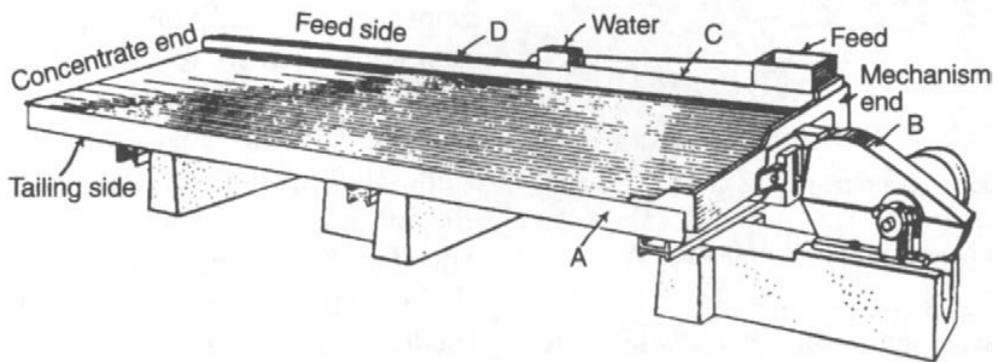


Figure 2.4 Shaking table (Wills, 1985)

It consists of a slightly inclined deck, A, on to which feed, at about 25 % solids by weight, is introduced at the feed box and is distributed along C; wash water is distributed along the balance of the feed side from launder D. The table is vibrated longitudinally, by the mechanism B, using a slow forward stroke and a rapid return, which causes the mineral particles to "crawl" along the deck parallel to the direction of motion. The minerals are thus subjected to two forces, that due to the table motion and that, at right angles to it, due to the flowing film of water. The net effect is that the particles move diagonally across the deck from the feed end and, since the effect of the flowing film depends on the size and density of the particles, they will fan out on the table, the smaller, denser particles tiding highest towards

the concentrate launder at the far end, while the larger lighter particles are washed into the tailings launder, which runs along the length of the table. Figure 2.5 shows an idealized diagram of the distribution of table products. An adjustable splitter at the concentrate end is often used to separate this product into two fractions, a high-grade concentrate and a middlings fraction.

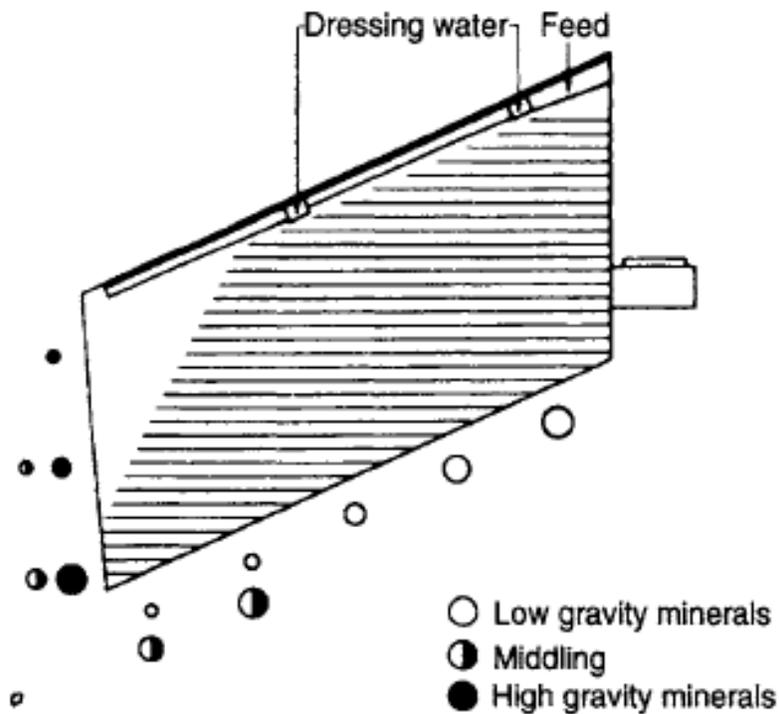


Figure 2.5 Distribution of shaking table products (Wills, 1985)

Although true flowing film concentration requires a single layer of feed, in practice a multilayered feed is introduced on to the table, enabling much larger tonnages to be dealt with. Vertical stratification due to shaking action takes place behind the *riffles*, which generally run parallel with the long axis of the table and are tapered from a maximum height on the feed side, till they die out near the opposite side, part of which is left smooth (Figure 2.5). In the protected pockets behind the riffles the particles stratify so that the finest and heaviest particles are at the bottom and the coarsest and lightest particles are at the top (Figure 2.6). Layers of particles are

moved across the riffles by the crowding action of new feed and by the flowing film of wash water. Due to the taper of the riffles, progressively finer sized and higher density particles are continuously being brought into contact with the flowing film of water that tops the riffles. Final concentration takes place at the unriffled area at the end of the deck, where the layer of material is at this stage usually only one or two particles deep.

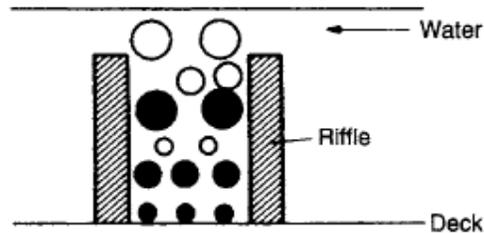


Figure 2.6 Vertical stratification between riffles (Wills, 1985)

The significance of the many design and operating variables and their interactions have been reviewed by (Sivamohan, 1985), and the development of a mathematical model of a shaking table is described by (Manser, 1991) The separation on a shaking table is controlled by a number of operating variables, such as wash water, feed pulp density, deck slope, amplitude, and feed rate, and the importance of these variables in the model development is discussed. Many other factors, including particle shape and the type of deck, play an important part in table separations. Flat particles, such as mica, although light, do not roll easily across the deck in the water film; such particles cling to the deck and are carried down to the concentrate discharge. Likewise, spherical dense particles may move easily in the film towards the tailings launder. The table decks are usually constructed of wood, lined with materials with a high coefficient of friction, such as linoleum, rubber, and plastics. Decks made from fiberglass are also used which, although more expensive, are extremely hard wearing. The riffles on such decks are incorporated as part of the mould. Particle size plays a very important role in table separations; as the range of sizes in a table feed increases, the efficiency of separation decreases. If a table feed

is made up of a wide range of particle sizes, some of these sizes will be cleaned inefficiently. In an idealized separation, the middlings produced are not "true middlings", i.e. particles of associated mineral and gangue, but relatively coarse dense particles and fine light particles. If these particles are returned to the grinding circuit, together with the true middlings, then they will be needlessly reground. Since the shaking table effectively separates coarse light from fine dense particles, it is common practice to classify the feed, since classifiers put such particles into the same product, on the basis of their equal settling rates. In order to feed as narrow a size range as possible on to the table, classification is usually performed in multi-spigot hydrosizers, each spigot product, comprising a narrow range of equally settling particles, being fed to a separate set of shaking tables. A typical gravity concentrator employing shaking tables (Figure 2.7) may have an initial grind in rod mills in order to liberate as much mineral at as coarse a size as possible to aid separation. The hydrosizer products feed separate sets of tables, the middlings being reground before returning to the hydrosizer. Riffled tables, or *sand tables*, normally operate on feed sizes in the range 3 mm to 100 μm , and the hydrosizer overflow, consisting primarily of particles finer than this, is usually thickened and then distributed to tables whose decks have a series of planes rather than fifties and are designated *slime tables*.

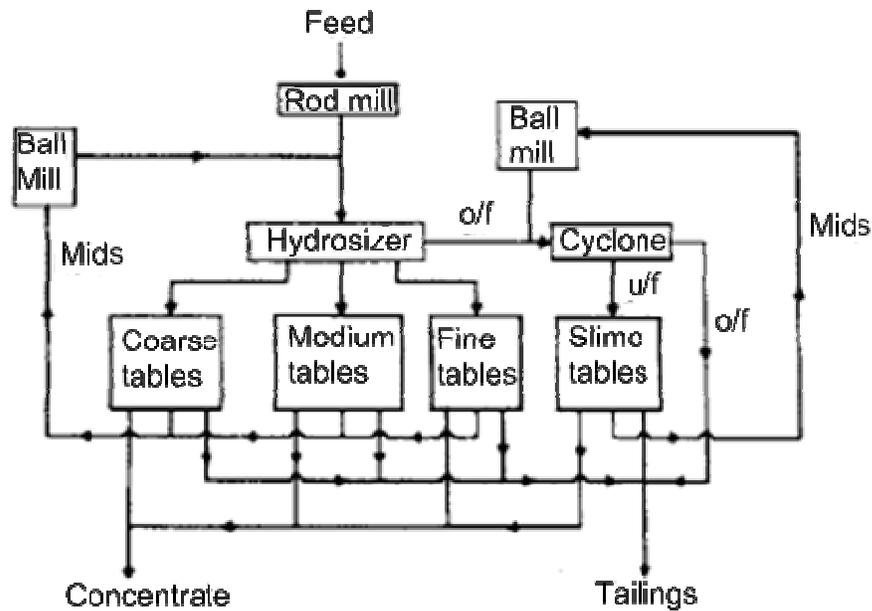


Figure 2.7 Typical shaking table concentrator flowsheet. (Wills, 1985)

Dewatering of the hydrosizer overflow is often performed by hydrocyclones, which also remove particles in the overflow smaller than about 10 μm , which will not separate efficiently by gravity methods due to their extremely slow settling rates. Successive stages of regrinding are a feature of many gravity concentrators. The mineral is separated at all stages in as coarse a state as possible in order to achieve reasonably fast separation and hence high throughputs. The capacity of a table varies according to size of feed particles and the concentration criteria. Tables can handle up to 2 t/h of 1.5 mm sand and perhaps 1 t/h of fine sand. On 100–150 μm feed materials, table capacities may be as low as 0.5 t/h. On coal feeds, however, which are often tabled at sizes of up to 15 mm, much higher capacities are common. A normal 5 mm raw coal feed can be tabled with high efficiency at 12.5 t/h per deck, whilst tonnages as high as 15.0 t/h per deck are not uncommon when the top size in the feed is 15 mm. (Terry, 1974). The introduction of double and triple-deck units (Figure 2.8) has improved the area/capacity ratio at the expense of some flexibility and control.

Separation can be influenced by the length of stroke, which can be altered by means of a hand wheel on the vibrator, or head motion, and by the reciprocating speed. The length of stroke usually varies within the range of 10–25 mm or more, the speed being in the range 240–325 strokes per minute. Generally, a fine feed requires a higher speed and shorter stroke which increases in speed as it goes forward until it is jerked to a halt before being sharply reversed, allowing the particles to slide forward during most of the backward stroke due to their built-up momentum. The quantity of water used in the feed pulp varies, but for ore tables normal feed dilution is 20–25% solids by weight, while for coal tables pulps of 33–40% solids are used. In addition to the water in the feed pulp, clear water flows over the table for final concentrate cleaning. This varies from a few liters to almost 100 l/min according to the nature of the feed material. Tables slope from the feed to the tailings discharge end and the correct angle of incline is obtained by means of a hand wheel. In most cases the line of separation is clearly visible on the table so this adjustment is easily made.

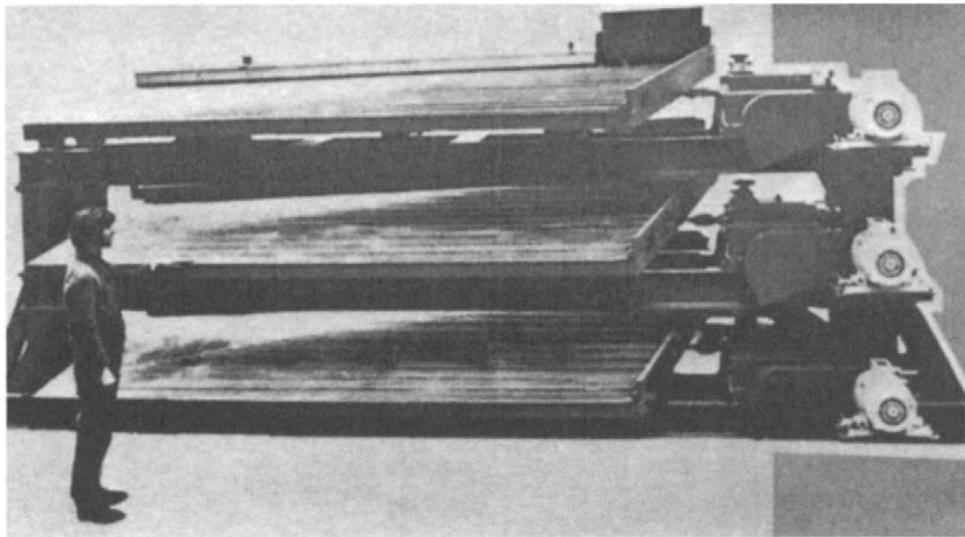


Figure 2.8 Triple-deck table (Wills, 1985)

The table is slightly elevated along the line of motion from the feed end to the concentrate end. The moderate slope, which the high-density particles climb more readily than the low-density minerals, greatly improves the separation, allowing much sharper cuts to be made between concentrate, middlings, and tailings. The correct amount of end elevation varies with feed size and is greatest for the coarsest and highest gravity feeds. The end elevation should never be less than the taper of the riffles; otherwise there is a tendency for water to flow out towards the riffle tips rather than across the riffles. Normal end elevations in ore tabling range from a maximum of 90 mm for a very heavy, coarse sand to as little as 6 mm for an extremely fine feed. Ore-concentrating tables are used primarily for the concentration of minerals of tin, iron, tungsten, tantalum, mica, barium, titanium, zirconium, and, to a lesser extent, gold, silver, thorium, uranium, and others.

2.1.4 Flotation

Froth flotation is probably the most important and versatile mineral processing method. The term ‘‘Flotation’’ replaced ‘‘Froth Flotation’’ by time and understood to represent the ‘‘Froth Flotation’’ process when used alone. The applicability of the method to a wide variety of ores and purposes, its adaptability according to varying ore and deposit characteristics, process economics, relatively simpler application, make flotation a distinguished technique in the mineral processing science and industry (Wills, 1985). The most important innovations, brought by flotation, are its capability to treat low grade ores that were once left as tailings and selective concentration of hard-to-process ores with complex natures. Use of a variety of reagents and development of new chemical agents, addressing very specific concentration needs, expanded the use of flotation from the treatment of metallic ores and industrial minerals to the cleaning of solid fuels in commercial scale. For instance, flotation was reported to be the most economic and effective means of fine coal preparation (-0.15 mm) on an industrial scale (Aplan, 1999).

Flotation is a three phase system for the separation of minerals according to their amenability to wetting. The schematic form of a flotation system is shown in Figure 2.9. It is based on the distinctions of the surface behaviors, i.e. the

differences in the physico-chemical characteristics of different mineral and/or metallic particles in a pulp. In a flotation pulp, mineral particles, air and water constitute the solid, gaseous and liquid phases, respectively (Figure 2.10). As has been mentioned, separation between particles of different identities takes place according to their affinity to wetting, i.e. their relative hydrophobic (water repellent) or hydrophilic (air-repellent) behaviors. Hydrophobic particles attach to the air bubbles and carried to the pulp surface in the form of a froth layer, while hydrophilic particles remain in the liquid phase of the flotation cell (Figure 2.9). The air bubbles should ideally have sufficient strength to lift the hydrophobic particles to the surface of the pulp and form the froth layer of certain thickness without breaking down. Therefore, materials with large size usually need comminution and size reduction to be successfully separated through flotation.

Generally, the maximum particle size is around 250 microns for metallic ores while this may be a little larger (500 microns) for coal.

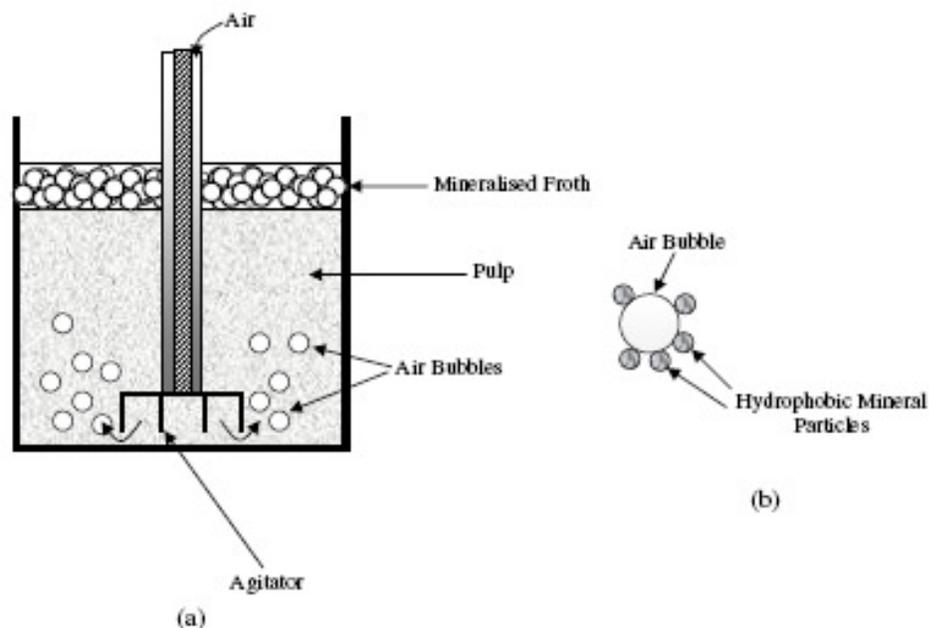


Figure 2.9 Schematic form of a) Flotation system b) Particle-bubble attachment (Wills, 1985)

The distinction of the surface properties, which is the fundamental requirement for flotation, can be further enhanced by use of a variety of flotation reagents. Such a modification is mostly required for increasing the effectiveness and efficiency of the process. This is accomplished by use of a number of specific chemical reagents (surfactants) that perform certain functions when added to a flotation pulp. These surfactants are classified as collectors, frothers and regulators. Collectors render target minerals hydrophobic by adsorbing on their surfaces through a variety of mechanisms, so that the particles with their modified water repellent surfaces may attach to the air bubbles and be recovered as the froth phase. If the valuable particles possess hydrophobic character and recovered as the froth phase, the process is called Direct Flotation. Sometimes it is more practical to leave the valuable particles in hydrophilic form, thus, in the pulp and to recover gangue or undesired entities as the froth phase. This process is called Reverse Flotation. Once the target minerals attach to the air bubbles, the bubble stability is of crucial importance for a complete removal of the attached particles out of the flotation system. Ideally, frother functions to reduce the surface tension at the liquid-vapor interface, without affecting the surface state of the minerals. This allows the generation of a higher amount of bubbles with smaller sizes and increased stability (Naik et. al., 2005). Regulator or modifier is the common name given to depressants, activators and pH regulators. Generally speaking, regulators are used to modify the collector action, i.e. by increasing or decreasing the effectiveness of the collector through various means, so that the water-repellent action of the collector is further adjusted to be specifically selective towards certain minerals. Activators are the reagents that alter the chemical characteristics of the mineral surface in order to render them hydrophobic or increase their affinity towards collector action. Depressants function in the opposite way and render certain mineral particles hydrophilic and prevent their flotation. pH regulators are used to adjust the pH of the flotation pulp to provide the most suitable acidity or alkalinity conditions to intensify the collector actions and surface characteristics in complex separations.

Contact angles and surface forces are probably the simplest way to express the theory of froth flotation. The hydrophobic or hydrophilic behavior of a mineral in the flotation pulp relies on the forces that act on its surface. Contact angles may describe the attaching forces between the solid phase (mineral) and gaseous phase (air bubble) as in Figure 2.10. The surface forces, which are also called as surface tension, result in the formation of a contact angle between the solid phase (mineral surface) and gaseous phase (air bubble). At equilibrium condition,

$$\gamma_{S/A} = \gamma_{S/W} - \gamma_{W/A} \cos \theta \quad (2.1)$$

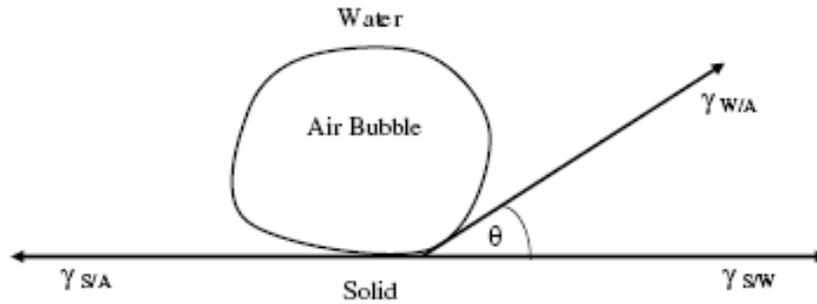


Figure 2.10 System of forces acting on a particle in the flotation pulp and the contact angle. (Wills, 1985)

In this equation $\gamma_{S/A}$, $\gamma_{S/W}$ and $\gamma_{W/A}$ denote the surface energies between solid-air, solid-water and water-air phases, respectively. θ is the contact angle between the mineral surface and the air bubble. Adhesion work, which is the work required for breaking the interface between the particle surface and air bubble, can be expressed using the interfacial forces, as it is equal to the work needed to separate the solid-air interface resulting in distinct air-water and solid-water interfaces:

$$W_{S/A} = \gamma_{W/A} + \gamma_{S/W} - \gamma_{S/A} \quad (2.2)$$

When Eq. 2.1 is combined with Eq. 2.2, the following formula is obtained:

$$W_{S/A} = \gamma_{W/A} (1 - \cos \theta) \quad (2.3)$$

Eq. 2.3 shows that the attachment force between the mineral surface and the air bubble increases with an increase in the contact angle. In other words, the contact angle is one of the most important characteristics of the minerals. The higher the contact angle of a mineral is, the higher its floatability will be.

2.2 Previous Studies with Asphaltites

As mentioned earlier, asphaltite is not an abundant substance and because of this there are not many studies on this matter. The most important studies about asphaltite are compiled below.

Hiçyılmaz and Altun (2006) investigated processing of Şırnak asphaltite sample by gravity and flotation concentration methods to decrease its ash content. Finely disseminated inorganic constituents of asphaltite revealed the difficulty of operation. Ash content of asphaltite was reduced from 44.86% to 31.44% by gravity concentration method with a 75% combustible recovery. On the other hand, it was possible to reduce ash content to 24% by flotation with almost same combustible recovery. Table 2.2 shows the ash, total sulfur, calorific value, combustible recovery properties of raw and improved asphaltites by gravity concentration results of this investigation.

In another study Ayhan et al. (2003) investigated the possibility of cleaning Şırnak asphaltite by froth flotation. For this purpose, laboratory tests were carried out in order to investigate the effects of grinding time, pulp density, pH, collector amount, depressant amount, frother amount on the overall performance of the flotation process. Chemical properties of asphaltite used was 0.46% moisture, 44.63% ash, 37.84% volatile matter, 6.02% total sulfur, 4796 kcal/kg net calorific value, 4987

kcal/kg gross calorific value. The best conditions determined were: pulp density: 5%, pH: 8, collector amount: 0 g/t, depressant amount: 150 g/t and frother amount: 100 g/t with a clean asphaltite assaying 32.95% ash with 76.61% combustible yield.

Table 2.2 Properties of raw and improved asphaltites by gravity concentration

Sample	Ash (%)	Total Sulfur(%)	Calorific value (kcal/kg)	Combustible recovery (%)	Operation condition
Raw	44.86	5.53	4380	-	-
Jig conc.	39.76	4.72	4764	77.53	-9.53 mm. sample
Reichert sp. conc.	37.56	4.23	4919	70.02	-0.6 mm. sample
Reichert sp. conc.	35.16	3.78	5128	78.41	-0.3 mm. sample
Humphreys sp. conc.	31.44	3.01	5440	75.84	-0.3 mm. sample
Shaking table conc.	36.10	4.08	5010	94.47	-0.3 mm. sample

Abakay et al. (2004) investigated the possibility of cleaning Şırnak asphaltite by the agglomeration method. Experimental studies were conducted on the asphaltite sample with ash, sulfur, volatile matter and fixed carbon contents of 44.8, 6.0, 38.0, and 17.2 wt%, respectively. For this purpose, laboratory tests were carried out to investigate the effects of grinding time, bridging liquid type, bridging liquid concentration, pulp density and agitator speed on the overall performance of the agglomeration process. Furthermore, the effects of sea water and lake water on the combustible recovery and ash contents of the agglomerated products were investigated. The optimum conditions determined were: grinding time: 50 min, bridging liquid type: 50% kerosene: 50% fuel oil, bridging liquid concentration:

30%, pulp density: 12.5%, stirring speed: 1800 rpm. It was found that the effects of sea water and lake water on the removal of ash were negative.

Doymaz et al. (2003) aimed to decrease ash amount and to increase calorific value of Silopi asphaltites with using flotation and demineralization treatments. Chemical analysis results of raw asphaltite was 0.64% moisture, 34.35% ash, 28.79% volatile matter, 6.34% total sulfur, 30,000 kJ/kg net calorific value. -250 μ m asphaltite samples first subjected to flotation, then the samples were treated with NaOH and various acid solutions such as HCl, HNO₃, H₂SO₄ and HF. The best results are 40.50% ash removal and 35,550 kJ/kg of calorific value with experiment done 5% NaOH+40% HF.

Another investigation made by Doymaz et al. (2007). In this study, high mineral matter (ash) asphaltites from Silopi were demineralised using aqueous sodium hydroxide followed by various mineral acids such as HNO₃, HCl, H₂SO₄, and HF. The extraction with 5% NaOH followed by leaching with 10% H₂SO₄ plus 40% HF was the optimum approach for chemical cleaning of the asphaltite and the maximum degree of the demineralization was found as 59.56%. At the same time, the calorific value increased from 30 MJ/kg to 36.26 MJ/kg and ash content decreased from 34.35 % to 13.89% according to the original sample.

Majumder et al. (2007) studied cleaning of asphaltite fines with MGS. The results obtained from this study revealed that among other parameters studied, drum rotation and feed solids concentration play dominating roles in controlling the yield and ash contents of the clean asphaltite. According to this study MGS could produce a clean asphaltite with an ash content of 14.67% and a yield of 71.23% from a feed asphaltite having an ash content of 24.61%.

Saydut et al. (2007) studied the method of desulfurization and demineralization by aqueous caustic leaching (ACL) for treatment of asphaltite samples. The effect of different parameters such as alkali concentration, time and temperature on leaching efficiency. The concentration of caustic was varied from 0.1 to 1.0 M, temperature from 100 to 180 °C, and leaching time from 4 to 16 h. Elevation of alkali concentration, leaching temperature and prolongation of time increase the removal

of total sulfur and ash. As a result of ACL, ash content of asphaltite was reduced from 40.08 to 22.14%, and 75% of combustible was recovered. Total sulfur and volatile matter content was reduced from 6.74 to 2.49% and from 33.72 to 19.10%, respectively.

Önal (1978) studied removing sulfur from Şırnak asphaltites with high intensity wet magnetic separation method. He found that sulfur content of asphaltite can be decreased from 2.94% to 1.40% with 85.6 % separation efficiency.

Molten caustic leaching process is effective in reducing significant amounts of ash-forming minerals, pyritic sulfur, and organic sulfur from solid fossil fuels. The effect of leaching asphaltite samples from Seguruk and Harbul collieries of Şırnak and Silopi asphaltite fields with molten sodium hydroxide and followed by mild acid on demineralization and desulfurization was investigated by Duz (2005). For NaOH/asphaltite (1:1, w/w) at 90 min and 400 °C pyritic sulfur was decreased from 2.35% to 0, organic sulfur was decreased from 4.08 % to 1.2% and total sulfur was decreased from 6.74% to 1.20% , ash content was decreased from 40.08% to 12.16%, volatile matter was decreased from 33.72% to 10.21% for Seguruk asphaltite with 76.18% asphaltite yield. For NaOH/asphaltite (1:1, w/w) at 90 min and 400 °C pyritic sulfur was decreased from 1.50% to 0, organic sulfur was decreased from 5.25% to 1.54% and total sulfur was decreased from 7.02% to 1.54% , ash content was decreased from 32.49% to 10.32%, volatile matter was decreased from 46.74% to 9.90% for Seguruk asphaltite with 68.56 asphaltite yield.

CHAPTER 3

MATERIALS AND METHODS

3.1 Karatepe Asphaltite Sample

In this study, asphaltite taken from Karatepe vein of Şırnak deposit was used. The characteristics of samples were determined through mineralogical and proximate analysis. Mineralogical analysis was performed under optical microscope on thin section of sample. The mineralogical analysis indicated that, addition to asphaltite, the sample contained calcite, dolomite, clay minerals and framboidal pyrite particles with size of 4-5 microns as ash forming minerals. Figure 3.1 shows the distribution of pyrite and other minerals in polished section of asphaltite. The results of proximate analysis and calorific value of sample are given in Table 3.1

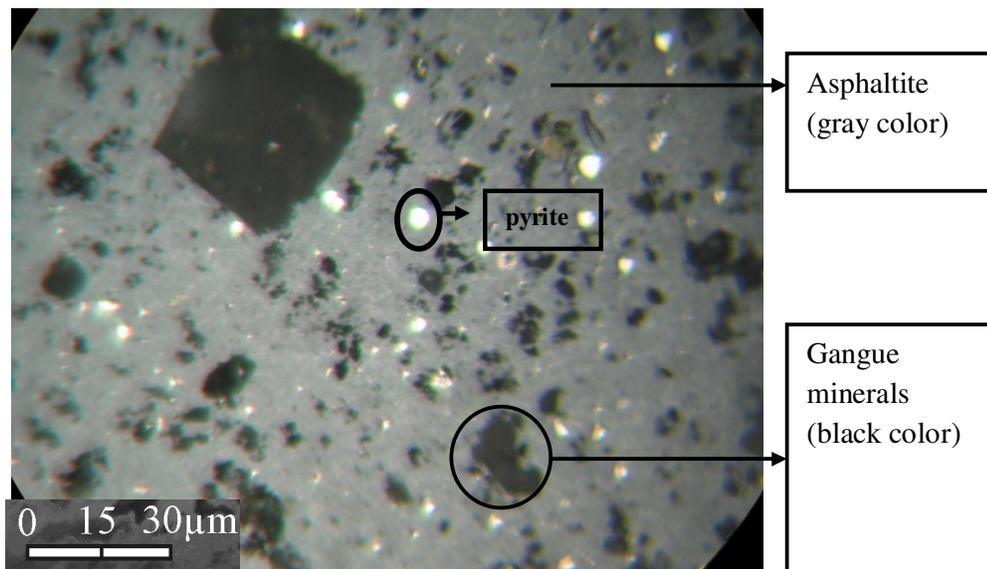


Figure 3.1 Framboidal Pyrites in Karatepe asphaltite samples.

Table 3.1 Proximate analysis of Karatepe asphaltite

Proximate Analysis	Weight (%) (as received)
Moisture	3.61
Ash	46.86
Volatile Matter	21.11
Fixed Carbon	22.42
Total Sulfur	5.56
Lower Calorific Value (kcal/kg)	3760
Upper Calorific Value (kcal/kg)	3910

3.2 Cleaning Studies

3.2.1 Shaking Table Studies

For shaking table studies -1000, -500 and – 250 μm asphaltite samples were used. Tests were carried out with laboratory type Wilfley shaking table.

3.2.2 Multi-Gravity Separator Studies

The Multi-Gravity Separator (MGS) experiments were carried out with Mozley C900 laboratory/pilot plant model MGS. During the tests effect of size were examined for -500, -250, -100 μm asphaltite samples.

3.2.3 Falcon Concentrator Studies

For Falcon concentrator studies laboratory type Falcon concentrator is used with -1000, -500, -250, -100 μm asphaltite samples, under 1, 2, 3, 4 psi water pressures and 30, 40, 50, 60, 68 and 70 Hertz frequency.

3.2.4 Flotation Studies

Flotation experiments were carried out with a laboratory type Wedag flotation machine with a cell of 650 ml nominal volume. During the flotation tests Aerosol 22 was used as wetting and dispersing agent, Accoal 9630 was used as collector, Dextrin was used as asphaltite depressants, Na_2SiO_3 was used as depressant for ash forming minerals. During flotation tests the effect of dosages of collector and depressants, feed size, pulp density, number of cleaning stages and ultrasonic treatment were examined.

3.3 Evaluation of Test Results

After each experiments ash content of products were determined. Ash analysis was done in accordance with the ASTM-D 3174-89 standard. Approximately 1g of dried representative sample was used for the ash analysis. The dried sample was heated in laboratory furnace from ambient temperature to 700 to 750°C in two hours. The sample was kept in the furnace for another two hours at 700 to 750°C. Then, the sample was removed and left to cool down in a dessicator and weighted. The results of the experiments were evaluated in terms of combustible recovery by using the following the formulas (Banerjee, 2003)

$$\text{Combustible Recovery} = \frac{(\% \text{ yield of clean asphaltite}) (100 - \% \text{ ash in clean asphaltite})}{(100 - \% \text{ ash in feed})} \quad (3.1)$$

CHAPTER 4

RESULTS AND DISCUSSION

4.1 Cleaning Studies with Shaking Table

For the separation of the ash forming minerals of the asphaltite sample, shaking table experiments were carried out with feed size of -1000, -500 and -250 microns and results were tabulated in Table 4.1

Table 4.1 Results of shaking table tests.

Feed Size	Products	Weight (%)	Ash (%)	Combustible Recovery (%)
-1000 microns	clean asphaltite	56,74	42,02	60,82
	middling	19,31	44,22	19,91
	tailing	23,95	61,87	16,88
-500 microns	clean asphaltite	46,98	41,16	51,11
	middling	26,53	44,26	27,34
	tailing	26,49	60,86	19,17
-250 microns	clean asphaltite	46,05	40,37	50,77
	middling	29,71	43,82	30,86
	tailing	24,24	64,32	15,99

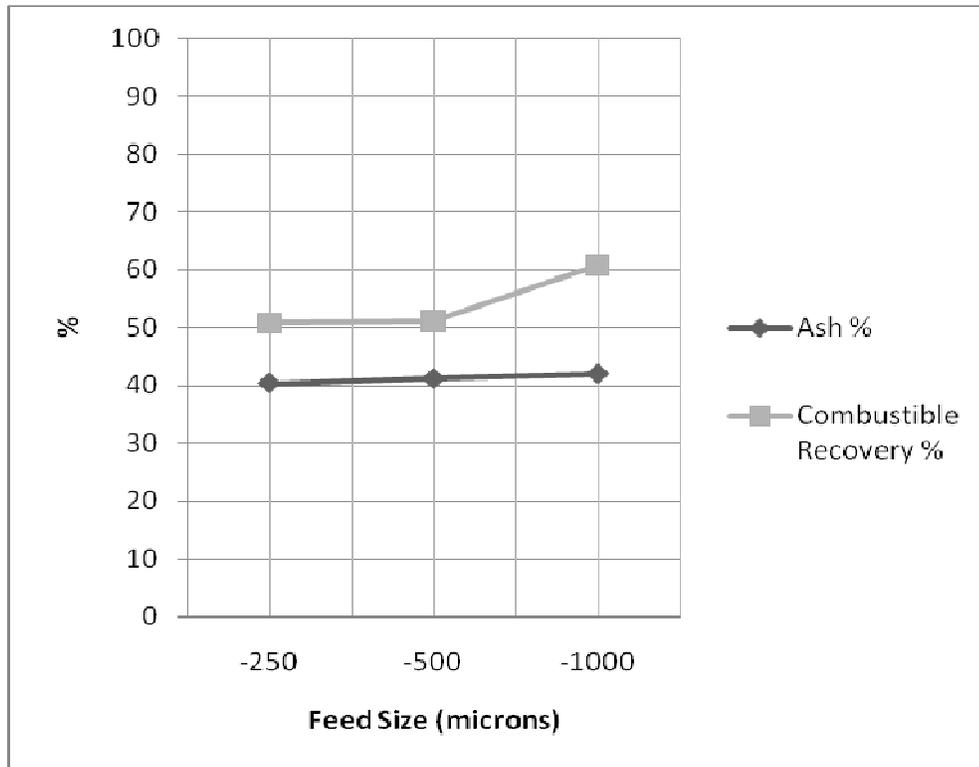


Figure 4.1 Shaking table results

The results of shaking table tests showed that cleaning of asphaltite with shaking table was not successful due to mineralogical texture of the sample. Ash forming minerals are very finely disseminated within the asphaltite matrix. This texture caused insufficient degree of liberation and decreased the specific gravity differences between the particles, therefore ash content of the sample could not be reduced sufficiently.

4.2 Cleaning Studies with Multi Gravity Separator

After preliminary tests with Multi Gravity Separator -100 microns feed size, 20% pulp density, 2 l/min feeding rate and 280 rpm drum speed were found as optimum

design and operating parameters. These parameters were kept constant during the tests which are performed to see the effect of wash water and tilting of drum.

The results of MGS tests are tabulated in Table 4.2. As shown in table the results were not successful. Due to coating of a few micron size ash forming minerals on asphaltite particle surface, ash content of sample could not be decreased sufficiently. Therefore, further tests were not carried out with Multi Gravity Separator.

Table 4.2 Results of Multi Gravity Separator tests

Exp No	Tilt (°)	Amplitude (mm)	Wash water (l/min)	Product	Weight (%)	Ash (%)	Combustible Recovery
1	2	20	3	clean asphaltite	83.06	43.30	88.94
				tailing	16.94	65.42	11.06
				feed	100.00	47.05	
2	2	10	3	clean asphaltite	81.23	43.53	86.74
				tailing	18.77	62.65	13.26
				feed	100.00	47.12	
3	0	20	3	clean asphaltite	81.25	43.77	86.51
				tailing	18.75	62.00	13.49
				feed	100.00	47.19	
4	0	20	1.5	clean asphaltite	77.06	42.56	83.84
				tailing	22.94	62.80	16.16
				feed	100.00	47.20	

4.3 Cleaning Studies with Falcon Concentrator

A series of cleaning tests were carried out with Falcon concentrator for different sizes of feed under different water pressure and speed.

Table 4.3. Effect of speed on ash content and combustible recovery of Falcon concentrator (feed size : -1000 microns, water pressure : 1psi)

Feed size	Water pressure (psi)	Speed (Hz)	Products	Weight %	Ash %	Combustible Recovery
-1000 microns	1	30	clean product	69.24	43.33	74.31
			tailing	30.76	55.91	25.69
-1000 microns	1	40	clean product	67.49	43.83	71.8
			tailing	32.51	54.2	28.2
-1000 microns	1	50	clean product	66.81	43.97	70.9
			tailing	33.19	53.7	29.1

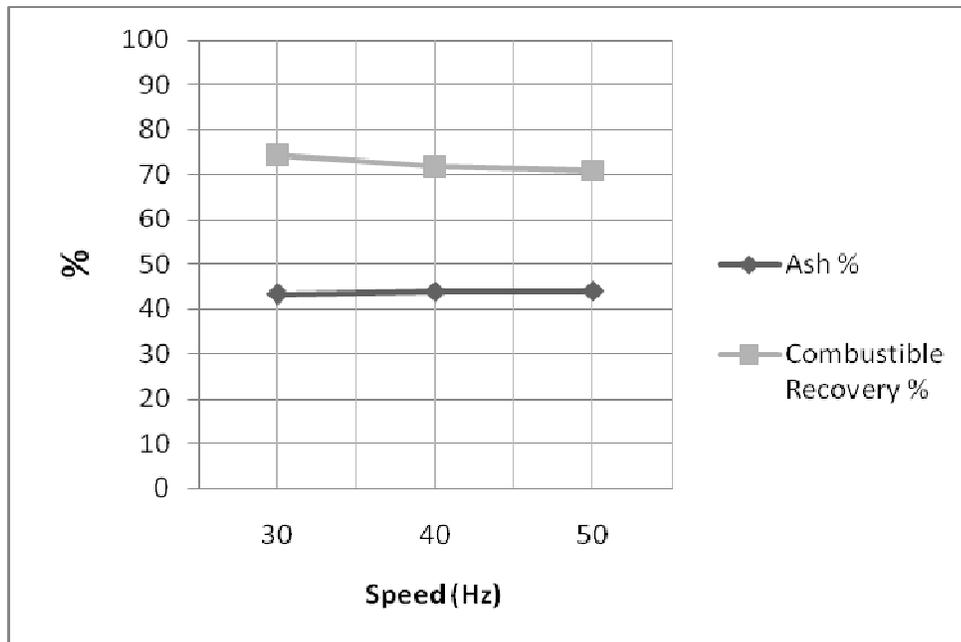


Figure 4.2 Effect of speed on ash content and combustible recovery of Falcon concentrator (feed size : -1000 microns, water pressure : 1psi)

Table 4.4 Effect of water pressure on ash content and combustible recovery of Falcon concentrator (feed size: -1000 microns, speed : 30Hz)

Feed size	Water pressure (psi)	Speed (Hz)	Products	Weight %	Ash %	Combustible Recovery
-1000 microns	2	30	clean product	68.01	44.11	71.99
			tailing	31.99	53.77	28.01
-1000 microns	3	30	clean product	68.72	44.17	72.66
			tailing	31.18	54.03	27.15
-1000 microns	4	30	clean product	69.11	44.52	72.62
			tailing	30.89	53.2	27.38

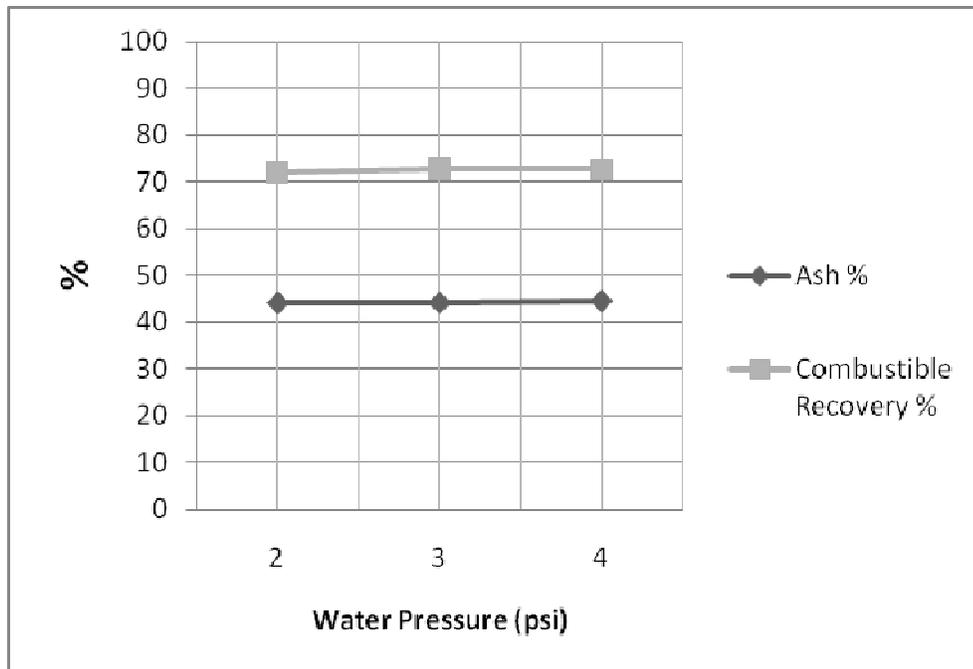


Figure 4.3 Effect of water pressure on ash content and combustible recovery of Falcon concentrator (feed size : -1000 microns, speed : 30Hz)

Table 4.5 Effect of speed on ash content and combustible recovery of Falcon concentrator (feed size: -500 microns, water pressure: 4 psi)

Feed size	Water pressure (psi)	Speed (Hz)	Products	Weight %	Ash %	Combustible Recovery
-500 microns	4	50	clean product	75.38	43.54	80.61
			tailing	24.62	58.41	19.39
-500 microns	4	60	clean product	63.81	43.72	68.02
			tailing	36.19	53.34	31.98
-500 microns	4	70	clean product	63.31	43.29	68
			tailing	36.69	53.95	32

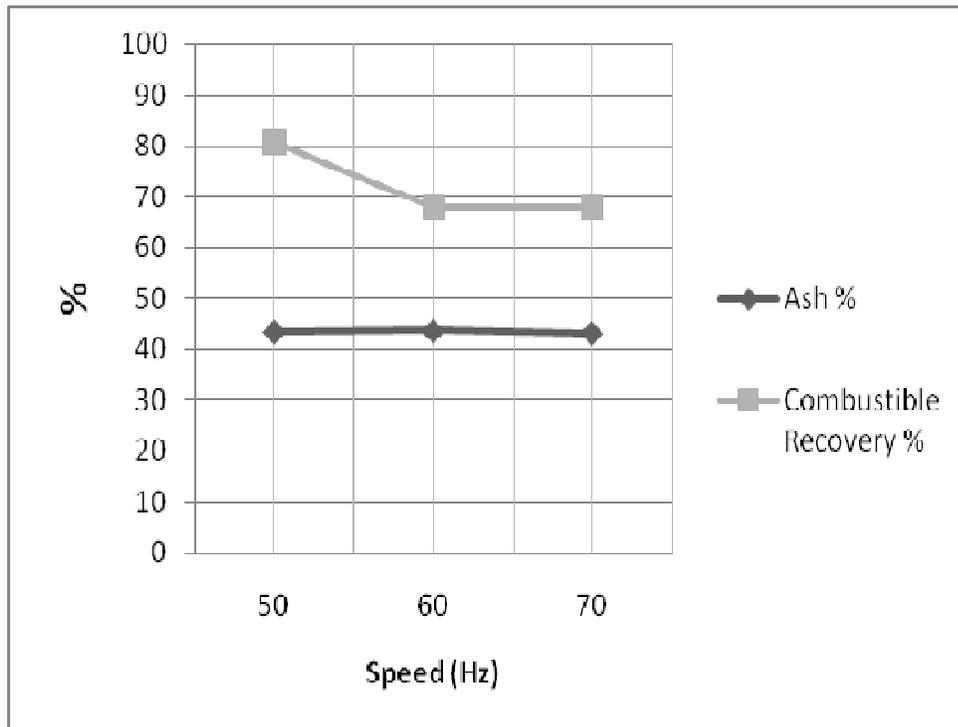


Figure 4.4 Effect of speed on ash content and combustible recovery of Falcon concentrator (feed size: -500 microns, water pressure: 4 psi)

Table 4.6 Effect of water pressure on ash content and combustible recovery of Falcon concentrator (feed size: -500 microns, speed: 50Hz)

Feed size	Water pressure (psi)	Speed (Hz)	Products	Weight %	Ash %	Combustible Recovery
-500 microns	1	50	clean product	68.18	42.71	73.98
			tailing	31.82	56.82	26.02
-500 microns	2	50	clean product	70.41	42.89	76.16
			tailing	29.59	57.46	23.84
-500 microns	3	50	clean product	74.91	43.48	80.19
			tailing	25.09	58.31	19.81

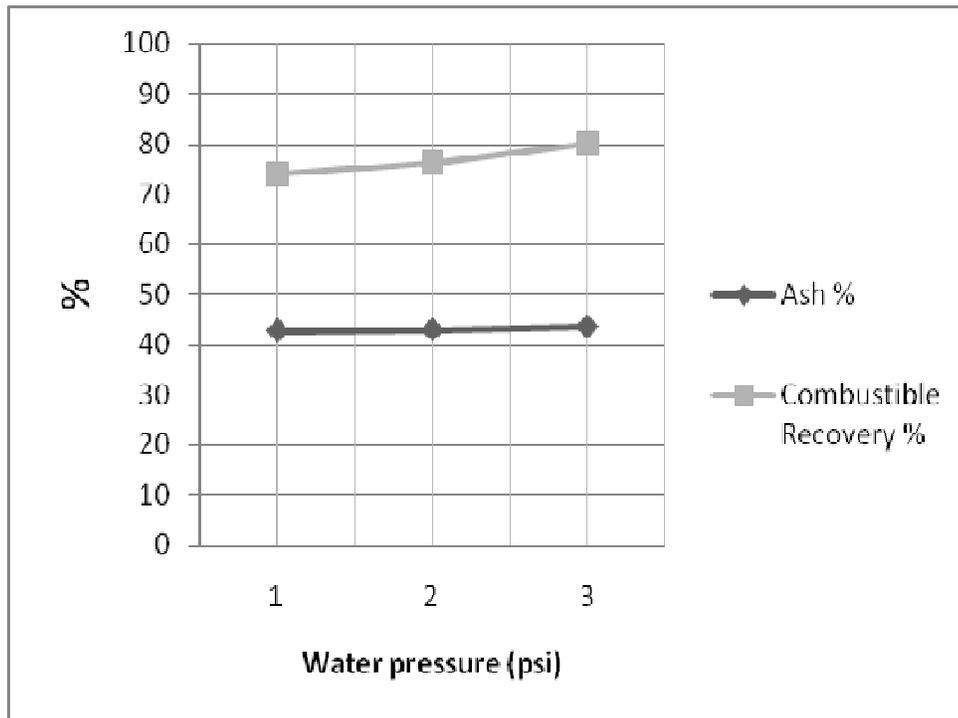


Figure 4.5 Effect of water pressure on ash content and combustible recovery of Falcon concentrator (feed size: -500 microns, speed: 50Hz)

Table 4.7 Effect of speed on ash content and combustible recovery of Falcon concentrator (feed size: -250 microns, water pressure: 4 psi)

Feed size	Water pressure (psi)	Speed (Hz)	Products	Weight %	Ash %	Combustible Recovery
-250 microns	4	50	clean product	74.21	42.45	80.89
			tailing	25.79	60.87	19.11
-250 microns	4	60	clean product	73.77	42.24	80.7
			tailing	26.23	61.15	19.3
-250 microns	4	70	clean product	73.54	42.11	80.63
			tailing	26.46	61.35	19.37

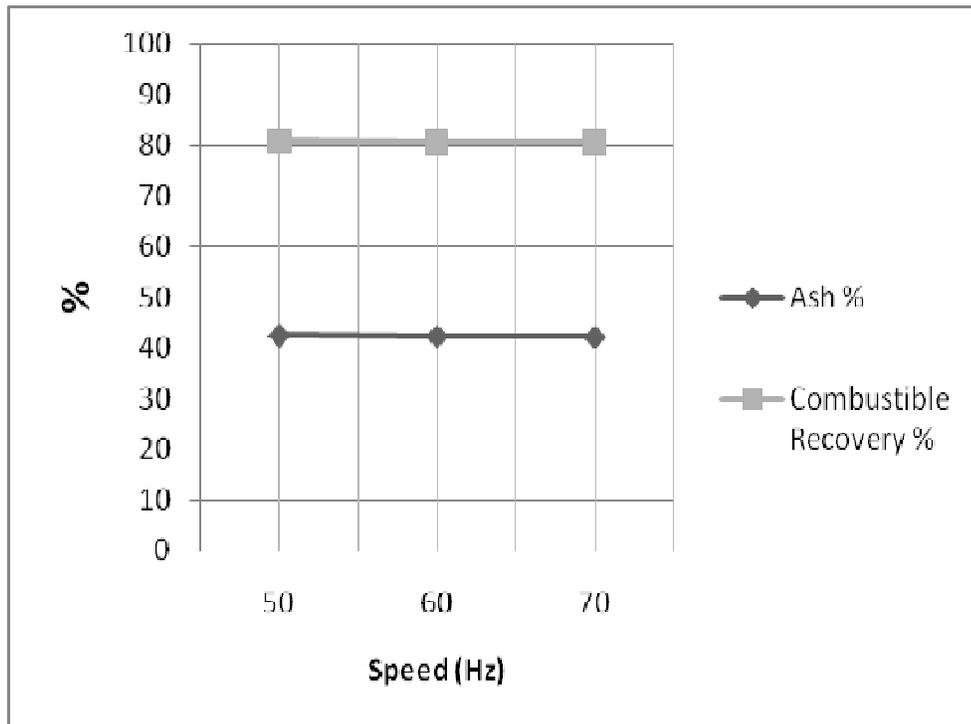


Figure 4.6 Effect of speed on ash content and combustible recovery of Falcon concentrator (feed size: -250 microns, water pressure: 4 psi)

Table 4.8 Effect of water pressure on ash content and combustible recovery of Falcon concentrator (feed size: -250 microns, speed: 60 Hz)

Feed size	Water pressure (psi)	Speed (Hz)	Products	Weight %	Ash %	Combustible Recovery
-250 microns	1	60	clean product	72.37	42.01	79.48
			tailing	27.63	60.79	20.52
-250 microns	2	60	clean product	72.91	42.15	79.88
			tailing	27.09	60.79	20.12
-250 microns	3	60	clean product	73.12	42.32	79.88
			tailing	26.88	60.47	20.12

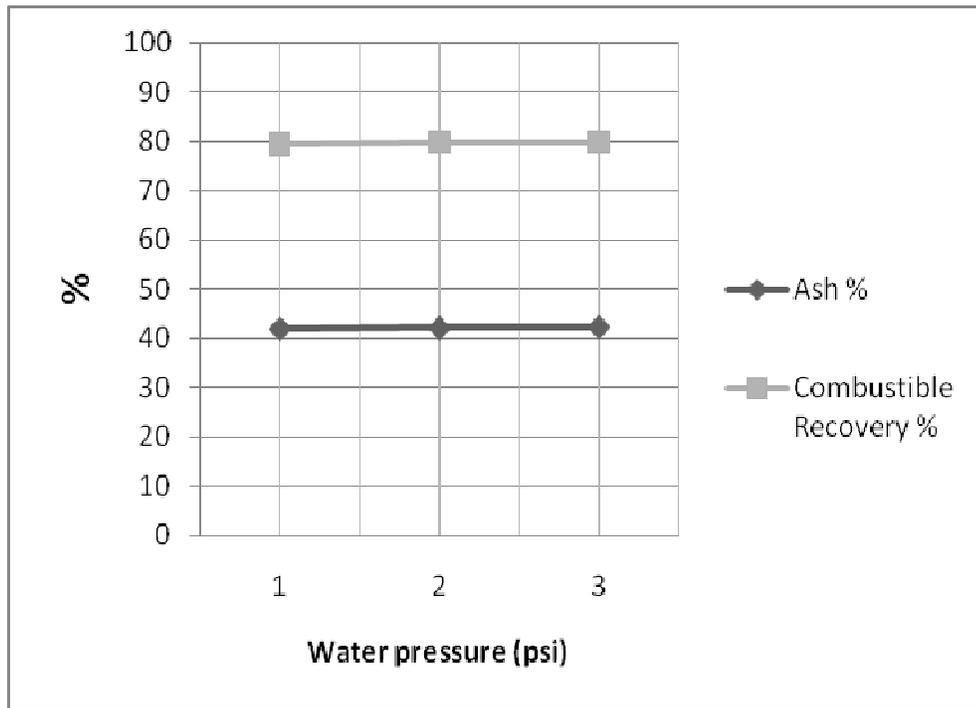


Figure 4.7 Effect of water pressure on ash content and combustible recovery of Falcon concentrator (feed size: -250 microns, speed: 60 Hz)

Table 4.9 Effect of water pressure on ash content and combustible recovery of Falcon concentrator (feed size: -100 microns, speed: 68 Hz)

Feed size	Water pressure (psi)	Speed (Hz)	Products	Weight %	Ash %	Combustible Recovery
-100 microns	4	68	clean product	78.47	41.01	87.67
			tailing	21.53	69.76	12.33
-100 microns	3	68	clean product	77.42	41.11	86.35
			tailing	22.58	68.08	13.65
-100 microns	1	68	clean product	76.81	41.82	84.64
			tailing	23.19	65.02	15.36

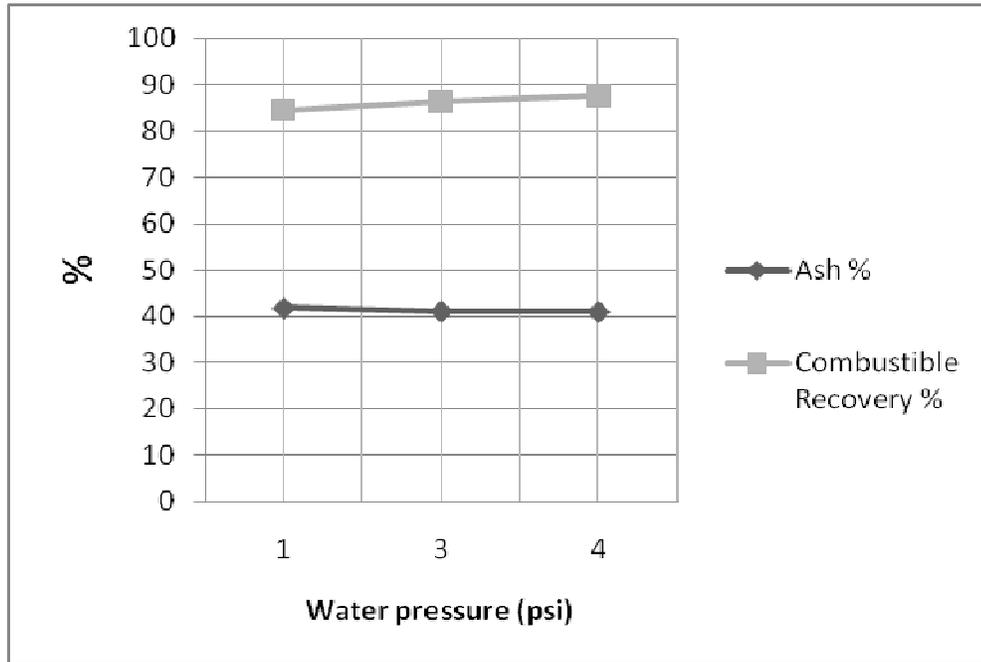


Figure 4.8 Effect of water pressure on ash content and combustible recovery of Falcon concentrator (feed size: -500 microns, speed: 68 Hz)

As seen from Tables and Figures, the clean products obtained by Falcon concentrator were rich in ash and the recovery of combustible was low. Most promising results were obtained for the – 100 micron feed size due to better liberation at this size. The best result was obtained at 4 psi water pressure and 68 Hz frequency with the yield of clean asphaltite 78.47%, the ash contents 41.01% and the combustible recovery 87.41%. Therefore cleaning and recleaning of valuable (light) product obtained under 4 psi water pressure and 68 Hz frequency (equivalent of 225 G acceleration) were tried. The flowsheets followed during cleaning and recleaning process are given in Figure 4.9 and Figure 4.10, and test results were tabulated in Table 4.10 and Table 4.11. Two stage cleaning with Falcon concentrator resulted in an asphaltite product with 37.68% ash content. There was a gradual decrease in ash content of product after each cleaning operation. But the reduction in ash is far from the specification required by Şırnak Municipality.

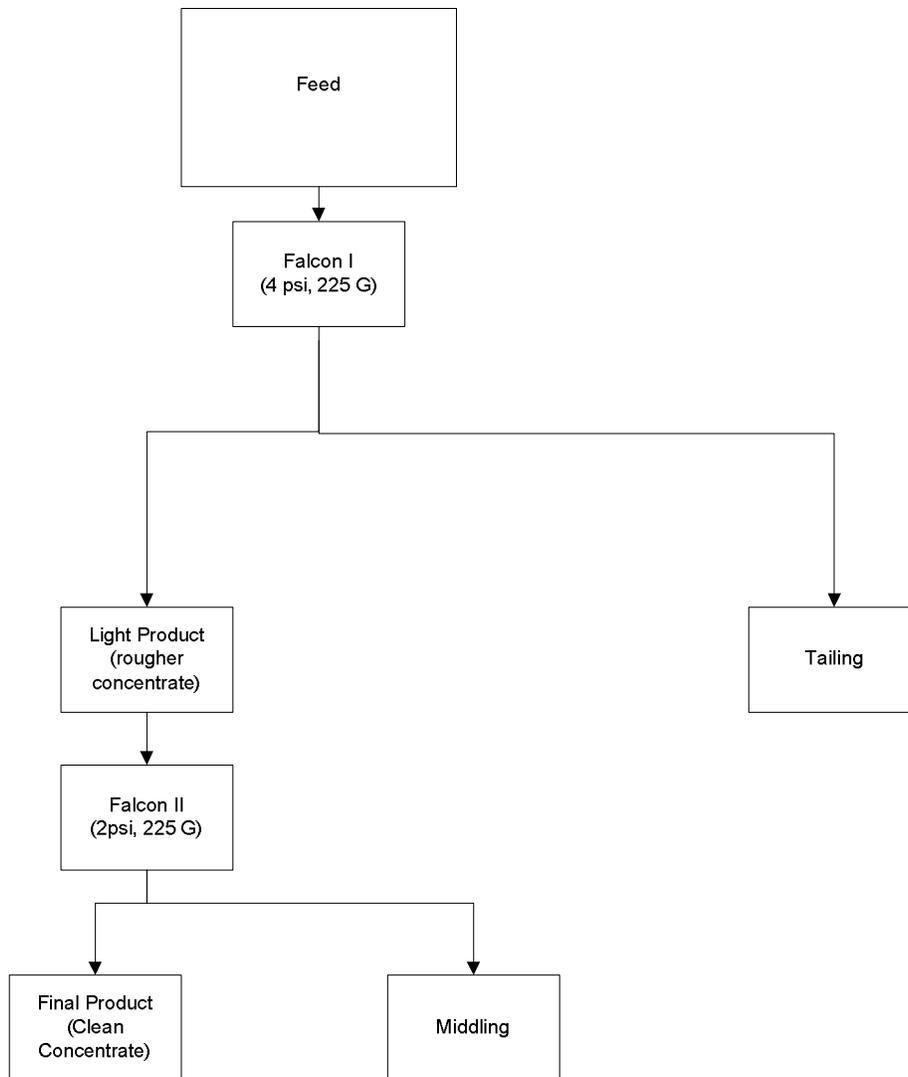


Figure 4.9 Single stage cleaning of rougher concentrate

Table 4.10 Results of single stage cleaning test with falcon concentrator

Products	Weight %	Ash %	Combustible Recovery %
Clean concentrate	73.31	39.53	84.12
Middling	5.49	66.09	3.54
Tailing	21.20	69.35	12.34
Feed	100.00	47.31	100.00

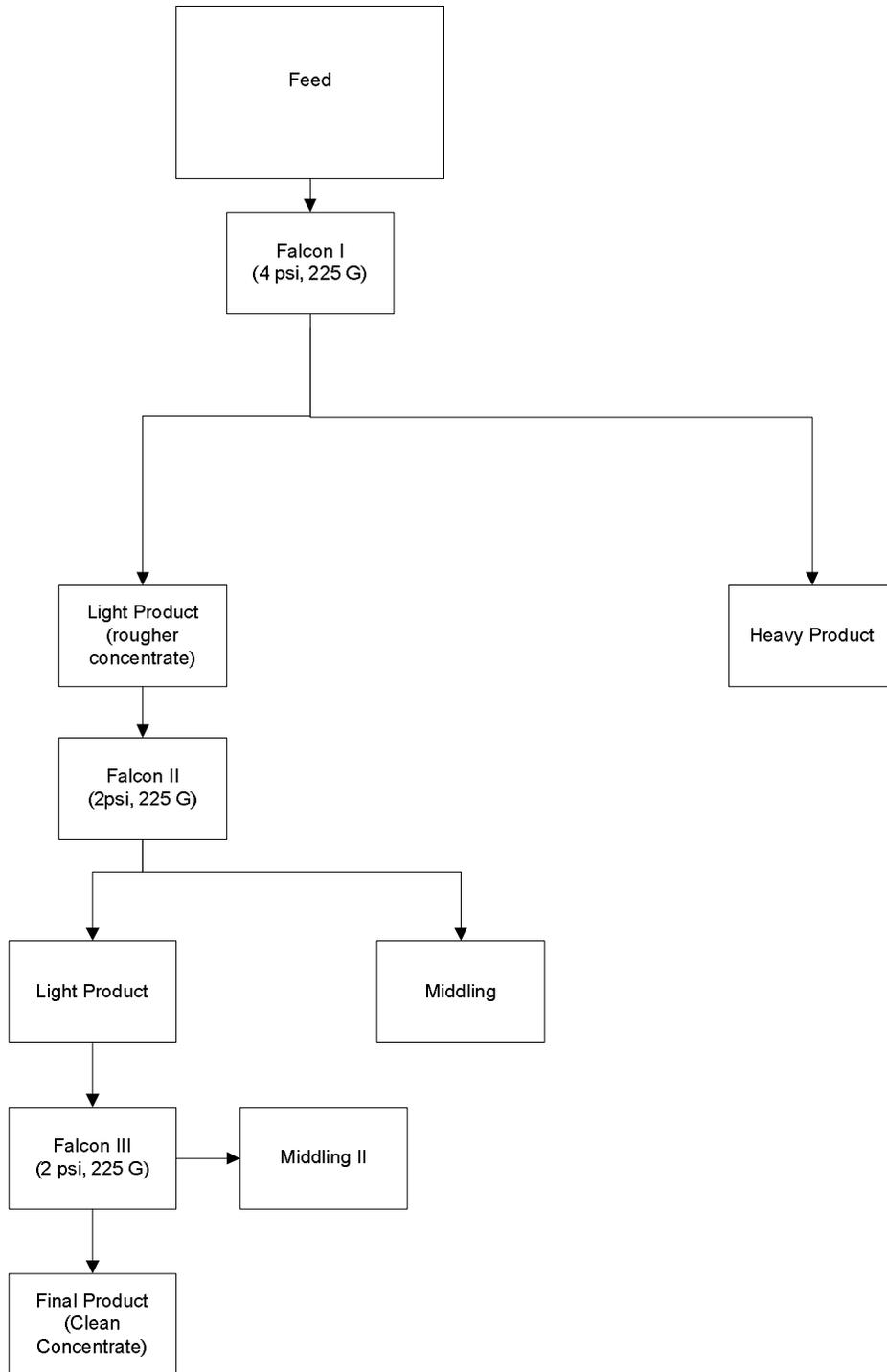


Figure 4.10 Two stage cleaning of rougher concentrate

Table 4.11 Results of two stage cleaning test with Falcon concentrator.

Products	Weight %	Ash %	Combustible Recovery %
Clean concentrate	69.77	37.68	81.87
Middling 1	6.81	67.33	4.19
Middling 2	1.51	52.04	1.36
Tailing	21.91	69.51	12.58
Feed	100	46.89	100.00

4.4 Cleaning Studies with Flotation

Due to increased environmental concerns associated with the use of fuel oil and kerosene as a collector, the industry has requested non-fuel oil collectors and in this study a new collector ACCOAL 9630 which is produced by CYTEC Industries Inc. was used.

4.4.1 Effect of Pulp Density on Asphaltite Flotation

Pulp density is one of the most important flotation parameter for asphaltite. This nature results from the inoculation of ash forming minerals within the asphaltite fines. Pulp density was changed from 10 to 20% solids by weight. During these tests no collector was used and tests were carried out with -1000 microns feed size at natural pH of the pulp. The results are tabulated in Table 4.12.

Table 4.12 Effect of pulp density on the collectorless flotation of Karatepe Asphaltite.

Pulp Density (%)	Products	Weight (%)	Ash (%)	Combustible Recovery (%)
20	Clean coal	18.89	39.90	22.47
	Tailing	81.11	48.48	77.53
15	Clean coal	15.19	31.94	22.01
	Tailing	84.81	49.65	77.99
10	Clean coal	6.64	28.75	9.88
	Tailing	93.36	49.20	90.12

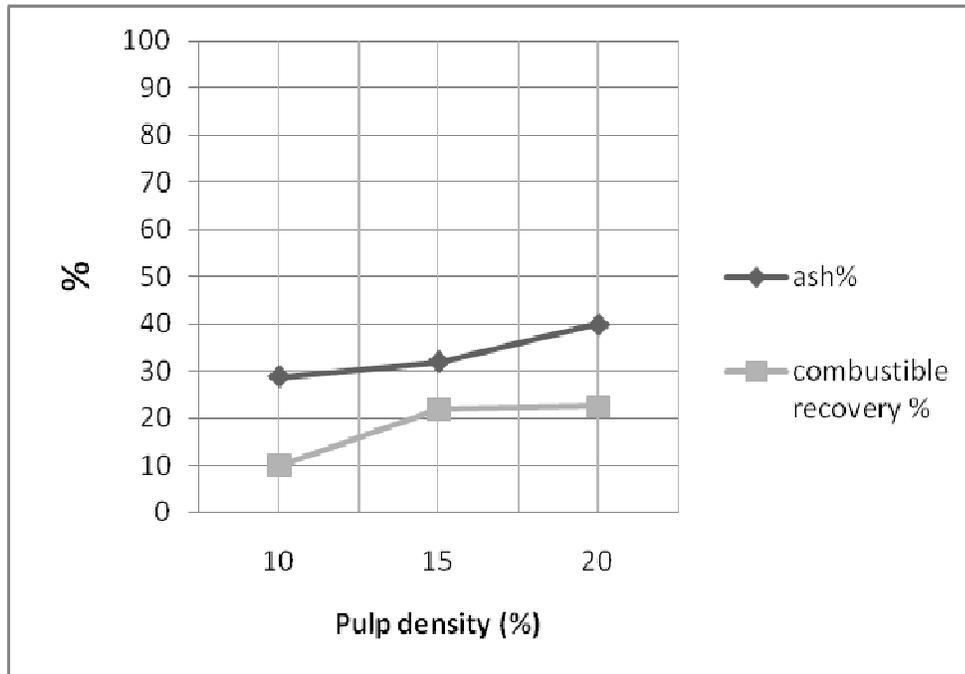


Figure 4.11 Effect of pulp density on the collectorless flotation of Karatepe Asphaltite.

As seen from the table, the ash % of concentrate significantly affected by the variation in the pulp density. Ash content of concentrate was decreased with the dilution of pulp. Concentrate with comparatively low ash content and with comperatively high combustible recovery was obtained at pulp density of 15% solid by weight.

4.4.2 Effect of Feed Size on Asphaltite Flotation

Collectorless flotation tests were also carried out to determine the effect of feed size on ash content and combustible recovery of concentrate. During these tests, asphaltite samples ground to 100% passing 75, 100, 250, and 500 microns were used. 15% solid by weight, 3 minutes of flotation time and natural pH of pulp were kept constant throughout these tests. The results were tabulated in Table 4.13.

Table 4.13 The effect of feed size on the collectorless flotation of Karatepe Asphaltite

Feed Size	Product	Weight %	Ash %	Combustible Recovery (%)
100% -500 micron	Concentrate	24.11	44.15	25.7
	Tailing	75.89	48.72	74.3
	Feed	100	47.61	100.00
100% -250 micron	Concentrate	25.15	43.86	26.96
	Concentrate	74.85	48.9	73.04
	Feed	100	47.63	100.00
100% -100 micron	Concentrate	35.81	41.41	39.99
	Concentrate	64.19	50.97	60.01
	Feed	100	47.54	100.00
100% -75 micron	Concentrate	36.06	42.32	39.79
	Concentrate	63.94	50.79	60.21
	Feed	100	47.74	100.00

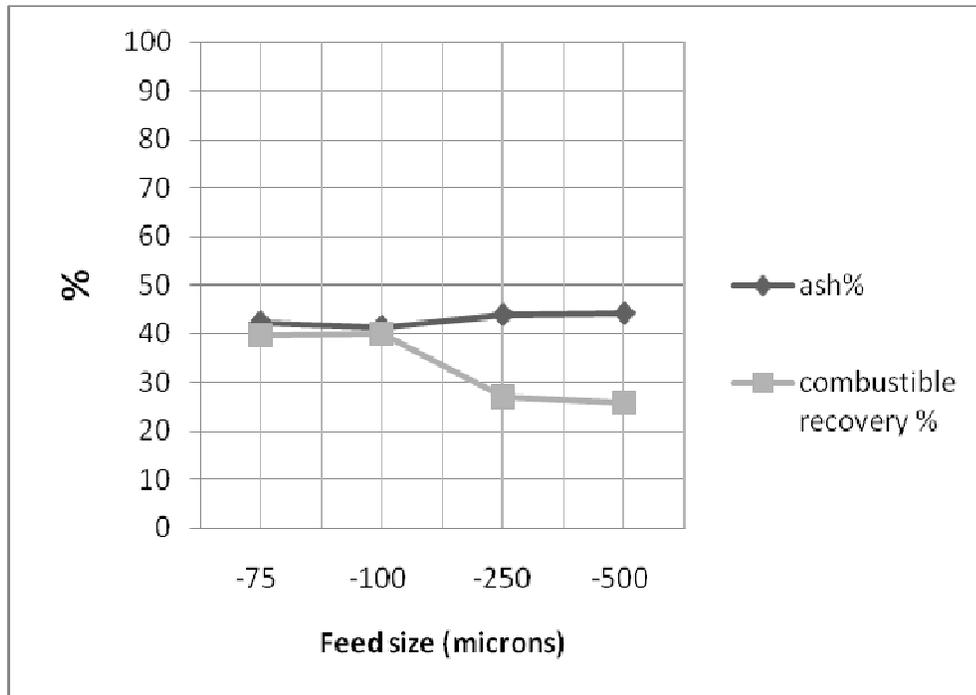


Figure 4.12 The effect of feed size on the collectorless flotation of Karatepe Asphaltite

As seen from Table 4.13, there is no further increase in combustible recoveries for finer than size 100% -100 microns. As the particle size decreased the coating of asphaltite particles with ash forming minerals was increased. Therefore size of feed was kept constant as 100% -100 microns for the following flotation test.

4.4.3 Effect of Collector Dosage on Asphaltite Flotation

Cleaning studies with Karatepe asphaltites were continued with flotation tests in the presence of collector namely Accoal 9630 which is the product of CYTEC Industries Inc. together with depressant 250g/ton Na_2SiO_3 . During these test Aerosol 22 which is also product of CYTEC Industries Inc. was used as wetting agent to prevent the agglomeration of very fine particles. Feed size, pH and pulp density were kept constant as -100 microns, natural pH and 10% solid by weight.

The results of the flotation experiments at different Accoal 9630 dosages are given in Table 4.14.

Table 4.14 Effect of Accoal 9630 addition amount on the flotation of Karatepe Asphaltite

Accoal (g/t)	Products	Weight (%)	Ash (%)	Combustible Recovery (%)
50	Concentrate	60.77	32.78	78.10
	Tailing	39.23	70.81	21.90
100	Concentrate	69.26	33.33	88.05
	Tailing	30.74	79.62	11.95
150	Concentrate	70.74	36.41	85.92
	Tailing	29.26	74.82	14.08

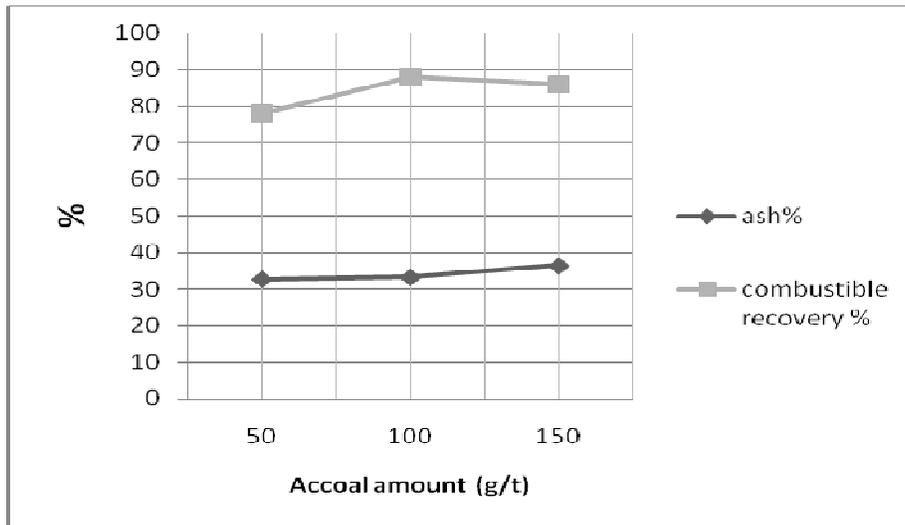


Figure 4.13 Effect of Accoal 9630 addition amount on the flotation of Karatepe Asphaltite

The lowest ash content (32.78) was obtained with the lowest collector dosage of 50 g/ton. However, the combustible recovery was also the lowest at this dosage (78.10 %). The combustible recovery increased to 88.05% with the 100g/ton Accoal 9630 addition with a very small increase in ash of concentrate.

4.4.4 Effect of Depressant on Asphaltite Flotation

A series of flotation tests were carried out to examine the effect of Na_2SiO_3 as depressant. During the test Accoal amounts, pH and feed size were kept constant as 100 g/ton, natural pH and -100 microns. The results were tabulated in Table 4.15.

Table 4.15 Effect of depressant (Na_2SiO_3) dosage on the flotation of Karatepe Asphaltite

Depressant (g/t)	Products	Weight (%)	Ash (%)	Combustible Recovery (%)
250	Concentrate	69.26	33.33	88.05
	Tailing	30.74	79.62	11.95
500	Concentrate	12.06	31.34	15.70
	Tailing	87.94	49.47	84.30
1000	Concentrate	7.52	29.34	12.52
	Tailing	92.48	49.05	87.48

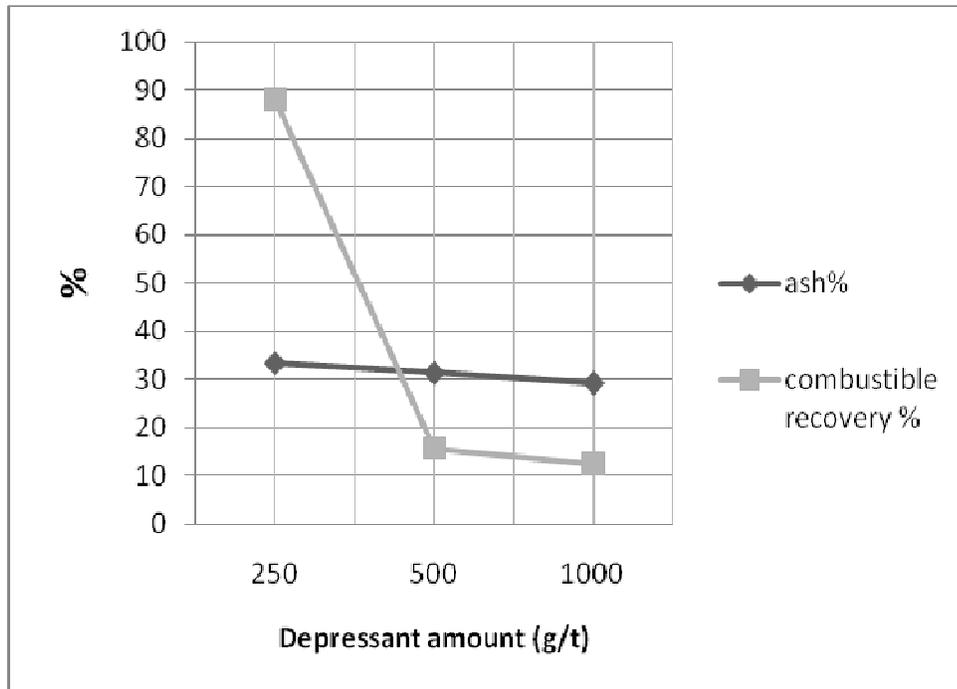


Figure 4.14 Effect of depressant (Na_2SiO_3) dosage on the flotation of Karatepe Asphaltite

As seen from Table 4.15 the combustible recovery of asphaltite is very sensitive to dosage of depressant. When depressant dosage was increased to 500 g/ton, ash content of concentrate decreased to 31.34% with a sharp decrease in combustible recovery. This indicates the negative effect of sodium silicate on combustible recovery, in other words, excess amount of Na_2SiO_3 depress the asphaltite particles also.

4.4.5 Cleaning of Rougher Asphaltite Concentrate

Cleaning of rougher concentrate was tried with stage addition of collector and depressant. The pulp density of 20% solid by weight was selected for the rougher flotation stage to maintain a reasonable pulp density during the cleaning stages. A large variety of flowsheets were followed and the promising flow sheets which are followed during the study are presented in Figures 4.15, 4.16 and 4.17. The results of these tests were tabulated in Table 4.16, 4.17 and 4.18

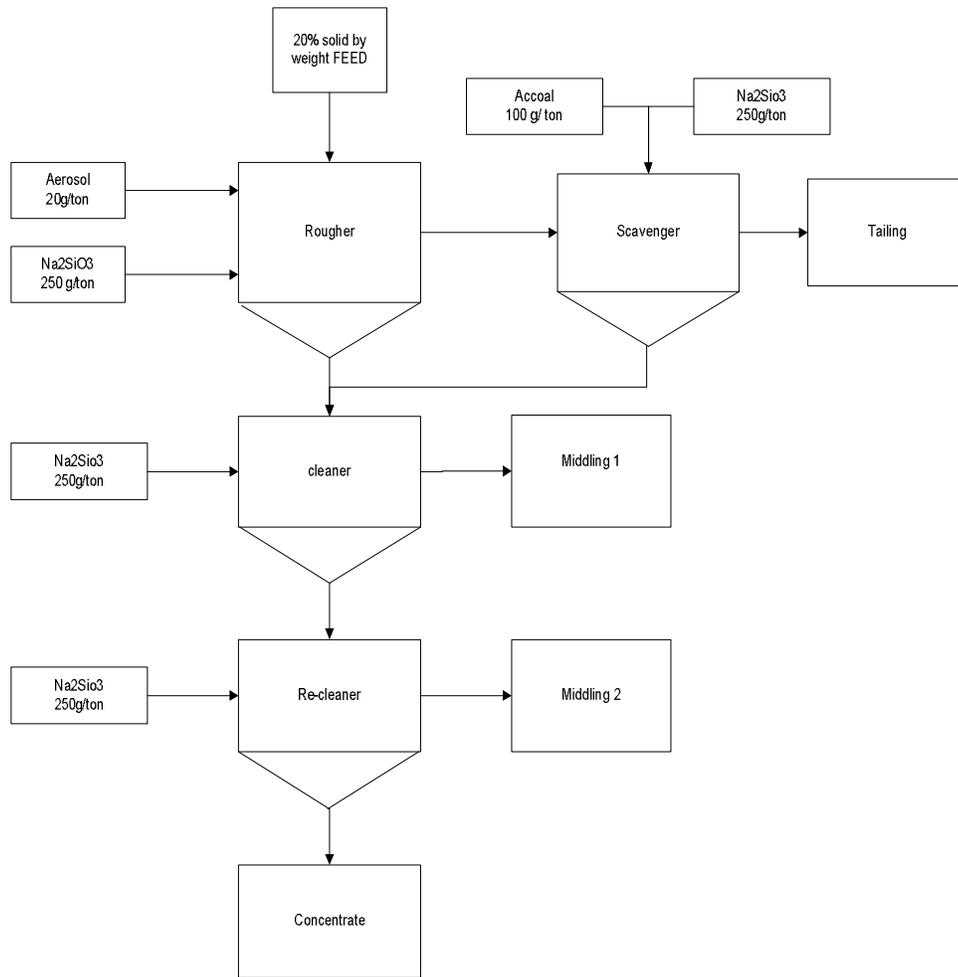


Figure 4.15 Two stage cleaning with stage addition of depressant.

Table 4.16 Results of two stage cleaning with stage addition of depressant.

Products	Weight %	Ash %	Combustible Recovery %
Concentrate	7.40	20.04	11.48
Middling 1	66.91	47.67	67.96
Middling 2	9.63	36.78	11.82
Tailing	16.06	72.07	8.71

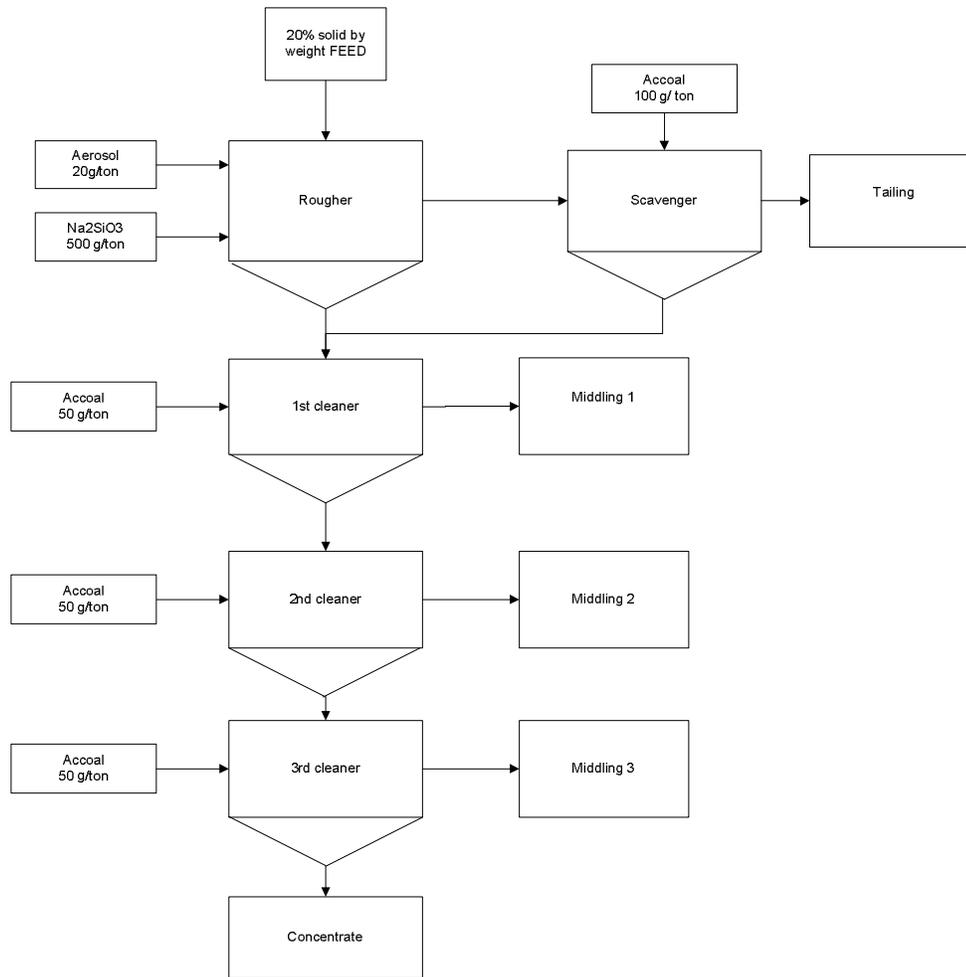


Figure 4.16 Three stage cleaning with stage addition of collector.

Table 4.17. Results of three stage cleaning with stage addition of collector.

Products	Weight %	Ash %	Combustible Recovery %
Concentrate	42.56	32.41	53.92
Middling 1	30.92	45.53	31.57
Middling 2	1.28	62.30	0.90
Middling 3	2.75	51.54	2.50
Tailing	22.49	73.67	11.10

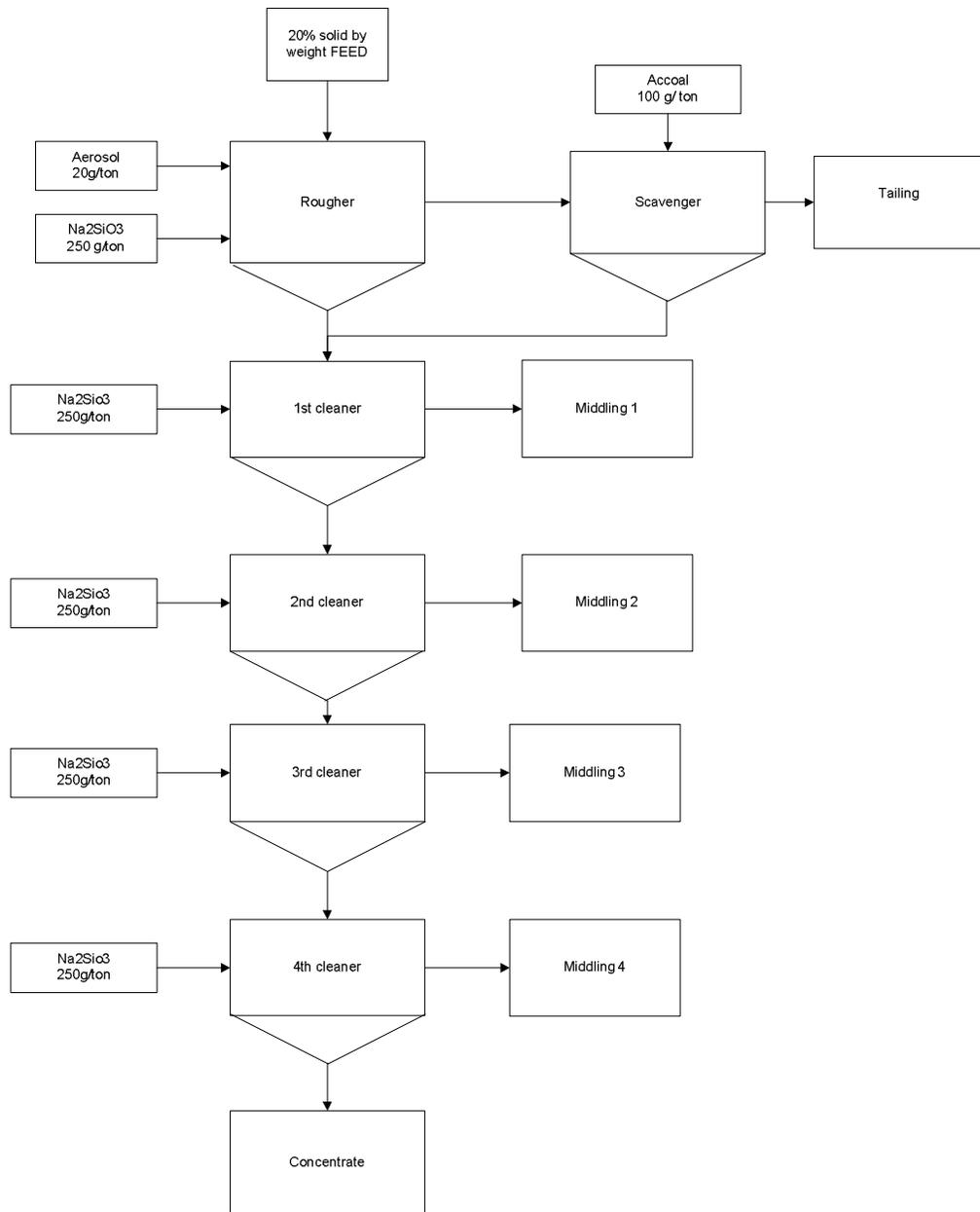


Figure 4.17 Four stage cleaning with stage addition of depressant

Table 4.18. Results of four stage cleaning with stage addition of depressant

Products	Weight %	Ash %	Combustible Recovery
Concentrate	12.96	21.02	19.36
Middling 1	9.79	59.30	7.53
Middling 2	32.61	45.49	33.61
Middling 3	22.16	41.65	21.45
Middling 4	8.17	32.91	10.37
Tailing	14.31	82.71	4.68

As seen from Table 4.16, the ash content of Karatepe asphaltite reduced from 48% to 20.04% with very low combustible recovery. The flotation of ash forming minerals together with asphaltite particle was the reason that prevents the efficient removal of ash forming minerals. On the other hand the depressive effect of Na_2SiO_3 on asphaltite was the reason of low combustible recovery.

4.4.6 Ultrasonic Assisted Flotation of Asphaltite

After the flotation of asphaltite with collector Accoal 9630 and depressant Na_2SiO_3 , the ash content of asphaltite was reduced to 20.04% with 11.48% combustible recovery. Physical interparticle attractive forces between asphaltite and ash forming minerals addition to surface coating of asphaltite particle with very fine ash forming minerals would be the other reason for high ash content and low combustible recovery. In this respect ultrasonic treatment of test sample was performed prior to and during the flotation experiments. During the ultrasonic assisted flotation test the flowsheet which is given in Figure 4.15 was followed. The obtained result was tabulated in Table 4.19

Table 4.19 The results of Ultrasonic Assisted Flotation Test

Products	Weight %	Ash %	Combustible Recovery %
Concentrate	9.22	17.59	15.31
Middling 1	49.91	41.46	53.82
Middling 2	9.67	35.08	11.79
Tailing	31.2	67.44	19.08

Ultrasonic treatment caused slight decrease in ash content of concentrate and the ash content decreased from 20.04% to 17.59%. On the other hand the combustible recovery of the concentrate increased from 11.48% to 15.31%. But these improvements in the ash content of concentrate and recovery of combustible was not enough to meet the market specification economically.

4.4.7 Reverse Flotation of Asphaltite with Cationic Collector

Cleaning of Karatepe asphaltite was also tested with reverse flotation in the presence of cationic type collector, namely Flotigam EDA which is a product of Clarient Inc. Tests are carried out at natural pH of the pulp, with 15% solid by weight pulp density and varying amounts of depressant dextrin with 100% -100 microns feed size. The results of cationic flotation tests were tabulated in Table 4.20.

Table 4.20. The effect of dextrin dosage on the reverse flotation of asphaltite.

Dosage of dextrin (g/ton)	Products	Weights %	Ash %	Combustible Recovery
50,00	Sink Product	60.57	55.33	52.00
	Froth Product	39.43	36.65	48.00
100,00	Sink Product	81.79	51.38	75.53
	Froth Product	18.21	29.25	24.47
150,00	Sink Product	90.21	49.41	86.82
	Froth Product	9.79	29.25	13.18
200,00	Sink Product	93.31	48.53	90.97
	Froth Product	9.69	28.74	9.03

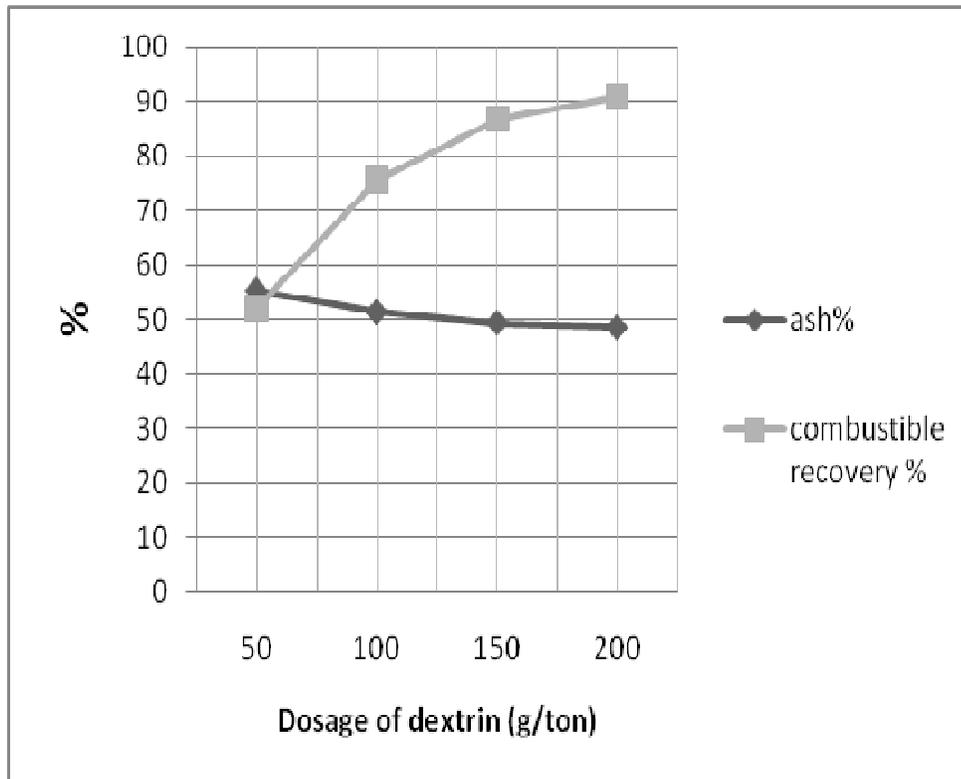


Figure 4.18. The effect of dextrin dosage on the reverse flotation of asphaltite.

The results tabulated in Table 4.20 revealed that the reverse flotation of asphaltite was not successful. The cationic collector Flotigam EDA added for flotation of ash forming minerals also floated the asphaltite particles, while the dextrin added for depression of asphaltite particles also depressed the ash forming minerals.

Shortly, selectivity was not maintained with cationic collector in the presence of depressant dextrin.

4.5 The Overall Evaluation of Cleaning Studies

The overall evaluation of cleaning studies of asphaltite with gravity concentration methods and flotation showed that it is not possible to produce a clean asphaltite product which meets the environmental regulations with respect to sulfur content.

The following table (Table 4.21) summarizes the optimum test conditions for each concentration method together with properties of obtained product.

Table 4.21 Summary of test results.

Concentration method	Operating conditions	Clean product			
		Ash %	S %	Lower calorific value	Combustible recovery %
Shaking Table	feed size : -250 microns	43.82	5.60	4550	50.77
Multi Gravity Separator	feed size : -250 microns pulp density: 20% solid Drum speed: 280 rpm Amplitude: 20 mm Tilting: 0°	42.56	5.65	4600	83.84
Falcon Concentrator	feed size : 100% -100 microns water pressure : 4 psi frequency: (equivalent of 225G) Two stage cleaning	37.68	5,85	4900	84.86
Flotation I	feed size : 100% -100 microns Two stage cleaning ultrasonic treatment assisted 20% solid by weight 100g/ton Accoal 250 g/ton Na_2SiO_3	17.59	6.68	6300	15.31
Flotation II	feed size : 100% -100 microns Natural pH 100g/ton Accoal 100 g/ton Na_2SiO_3	33.33	6,25	5250	88.05

CHAPTER 5

CONCLUSIONS

In the light of experimental results in this study, the following conclusions can be drawn;

- Karatepe asphaltite is not a clean fuel source for domestic heating due to its high ash and sulfur content of 46.86% and 5.56% respectively.
- Fine dissemination of ash forming minerals down to a few micron size requires very fine grinding to provide sufficient liberation.
- Very fine grinding reduced the effect of density difference between asphaltite and ash forming minerals during the gravity concentration and resulted low quality product with high ash content.
- Partial hydrophobicity of asphaltite and coating of slime size ash forming minerals on asphaltite particle and vice versa complicated the flotation process and decreased the efficiency of processes.
- Ultrasonic treatment enhanced the removal of slime size ash forming minerals from the asphaltite particle. Under ultrasonic treatment asphaltite product with 17.59% ash and 6.68% S content was obtained with 15.31% combustible recovery through the multi-stage cleaning. Eventhough this ash content of solid fuel is suitable for domestic heating, high sulfur content prevents the use of product as solid fuel for domestic heating.
- A research about the chemical demineralization of Şırnak Karatepe asphaltite is recommended.

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