

B. Tech thesis on

**EFFECT OF KEY VARIABLES ON
COAL CLEANING BY FROTH FLOTATION TECHNIQUE**

For partial fulfilment of the requirements for the degree of

Bachelor of Technology

in

Chemical Engineering

Submitted by:

Avilash Biswal

Roll Number- 108CH038

Under the guidance of:

Prof Pradip Rath



Department of Chemical Engineering,

National Institute of Technology,

Rourkela-769008

2012



CERTIFICATE

This is to certify that the thesis entitled “**Effect of Key Variables on Coal Cleaning by Froth Flotation Technique**” submitted by **Avilash Biswal (108CH038)** in partial fulfilment of the requirements for the award of degree of Bachelor of Technology in Chemical Engineering at National Institute of Technology, Rourkela is an authentic work carried out by him under my supervision and guidance.

To best of knowledge, the matter embodied in this thesis has not been submitted to any other university or institute for the award of any degree.

Date:

Place: Rourkela

Prof Pradip Rath

Department of Chemical Engineering,

National Institute of Technology,

Rourkela-769008

ACKNOWLEDGEMENT

I would like to convey my sincere gratitude to my project supervisor, Prof Pradip Rath for his invaluable suggestions, constructive criticism, motivation and guidance for carrying out related experiments and for preparing the associated reports and presentations. His encouragement towards the current topic helped me a lot in this project work which also created an area of interest for my professional career ahead.

I would like to thank Shri J. Nayak for his help during my experiments. I would also thank Shri S. Mohanty for arranging chemicals and accessories. I would also thank my friend Kiran Chandra Nayak for helping me in the initial part of this project work.

I owe my thankfulness to Prof R. K. Singh, Head, Department of Chemical Engineering for providing necessary facilities in the department and also to Prof H. M. Jena for the excellent coordination and arrangement towards the consistent evaluation of this project.

I thank my family and friends for their encouragement towards this project work.

Date:

Place: Rourkela

Avilash Biswal

Roll Number-108CH038

Department of Chemical Engineering,

NIT, Rourkela-769008

ABSTRACT

Coal, in India and Worldwide, is cleaned predominantly by dense medium separations and jigging. But when it comes to cleaning of fine coals such as mine dust, slack coal, washing rejects and slurries, froth flotation method of coal cleaning has better efficiency and handling. In this method of wet cleaning, froth is made in a flotation cell by bubbling air through water in presence of frothing agents; on addition, pure and light coal adhere to air bubble while the dirtier part sinks to bottom of the cell. Thus clean coal is recovered from the collected froth amount. Density and wettability can be termed as the two bases of separation which are the inherent properties of the coal. In this current project froth flotation was being carried in a fixed froth flotation cell used in the laboratory. Runs of froth flotation have been carried out using the same stock of coal but varying the size of coal fines, concentration of coal slurries and different doses of collecting agents and of their mixtures. In order to quantitatively measure the effect, ash rejection, combustible recovery and efficiency were calculated. Effect of size of particles in slurry was very much visible while the combustible recovery and ash rejection was enhanced when higher dose of collecting agents were used. Use of advanced frothing agents and pH controllers can be studied further in this direction.

Keywords: Coal cleaning, Froth flotation, Ash rejection, Combustible recovery

CONTENTS

Cover Page	
Certificate	i
Acknowledgement	ii
Abstract	iii
Contents	iv
List of Figures	vi
List of Tables	vii
Nomenclature	viii
1. Introduction	1
1.1 Coal and Its Utilization	1
1.2 Coal Cleaning	2
1.3 Advantages of Coal Cleaning	4
1.4 Current Study	4
2. Literature Review	5
2.1 Froth Flotation	5
2.2 Recent Trends and Developments	6
3. Materials and Methods	10
3.1 Sample Collection and Preparation	10

3.2	Proximate analysis	10
3.3	Washability Studies	12
3.3.1	Float and Sink Tests	12
3.3.2	Washability Curve	13
3.4	Froth Flotation Cell	14
3.5	Froth Flotation Tests	16
3.5.1	Constraints	16
3.5.2	Parameters in Current Study	16
3.5.3	Effect of Concentration of Slurry	17
3.5.4	Effect of Particle Size in Slurry	19
3.5.5	Effect of Collecting Agents and Doses	19
3.5.6	Calculations and Graphical Interpretation	20
4.	Results and Discussion	22
4.1	Proximate Analysis	22
4.2	Washability Studies	23
4.3	Effect of Concentration of Slurry	24
4.4	Effect of Particle Size in Slurry	27
4.5	Effect of Collecting Agents and Doses	29
4.6	Discussions	33
5.	Conclusion	37
	References	38

LIST OF FIGURES

Figure. No.	Title	Page No.
1	Classification of Coal Cleaning Processes	3
2	Float and Sink Test	13
3	Schematic Diagram of a Froth Flotation Cell	14
4	Photograph of Froth Flotation Unit in Laboratory	15
5	Image of froth at the onset of a run (t= 0 min)	18
6	Image of froth at the end of a run (t= 10 min)	18
7	Washability Characteristics Curve	24
8	% Ash Rejection vs. Concentration of Coal Slurry	25
9	% Combustible Recovery vs. Concentration of Coal Slurry	26
10	Efficiency Index vs. Concentration of Coal Slurry	26
11	% Ash Rejection vs. Particle Size in Coal Slurry	28
12	% Combustible Recovery vs. Particle Size in Coal Slurry	28
13	Efficiency Index vs. Particle Size in Coal Slurry	29
14	% Ash Rejection vs. Collecting Agents and Doses	32
15	% Combustible Recovery vs. Collecting Agents and Doses	32
16	Efficiency Index vs. Collecting Agents and Doses	33

LIST OF TABLES

Table No.	Title	Page No.
1	Observations of Proximate Analysis	22
2	Results of Proximate Analysis	22
3	Composition of Separating Medium (of 200 mL) for Float and Sink Test	23
4	Washability Data	23
5	Data of Froth and Tailings-1	24
6	Calculations for Effect of Concentration of Coal Slurry	25
7	Data of Froth and Tailings-2	27
8	Calculations for Effect of Particle Size in Coal Slurry	27
9	Data of Froth and Tailings-3 [using Mixture of Oils]	29
10	Data of Froth and Tailings-4 [using Kerosene]	30
11	Data of Froth and Tailings-5 [using Diesel]	30
12	Calculations for the Effect of Collecting Agents and Their Doses	31

NOMENCLATURE

$\% AR$	% Ash Rejection
$\% CR$	% Combustible Recovery
A_c	% Ash in Clean Coal
A_f	% Ash in Feed Coal
A_r	% Ash in Tailings
C	Concentration of Coal Slurry (g/L)
D	Doses of Collecting Agent (g/kg of feed)
EI	Efficiency Index
W_c	Weight of clean coal (g)
$W_{c,daf}$	Weight of Clean Coal, dry-ash free basis (g)
W_f	Weight of Feed Coal (g)
$W_{f,daf}$	Weight of Feed Coal, dry-ash free basis (g)
W_r	Weight of Tailings (g)
X	Particle Size in the Coal Slurry (μm)

CHAPTER – 1

INTRODUCTION

1. INTRODUCTION

1.1 COAL AND ITS UTILIZATION:

Coal is the most abundant fossil fuel available on the Earth which accounts for about 55 % of the World's electricity generation and the around 65 % in India. It can be defined as a complex heterogeneous mixture of plant substances which are altered due to physical and chemical processes. These processes have been taking place for several million years and have been accomplished by bacteria, heat and pressure inside the Earth's crust. It primarily consists of Carbon along with Hydrogen, Oxygen, Sulphur... etc. as secondary components.

[1][3]

Coal formation starts from the plant debris and ends at Graphite at its highest maturity. This process may be complete or may be stopped at any stage giving rise to coal of varying maturity thus various Ranks. The commonly used ranks of coal are Peat, Lignite, Sub-Bituminous, Bituminous, Semi-Anthracite and Anthracite. Peat, being the poorest variety, is usually not considered as coal. With the progress of coal formation, the amount of moisture and oxygen decreases; the amount of carbon increases. The calorific value increases from 4500 kcal/kg for Lignite to 8500 kcal/kg for Bituminous.

Apart from electricity generation in thermal power plants as stated above, coal finds its application in many other fields such as Metallurgical Coke Manufacture (in Steel

industries), Polymers, fibers, dyes, Alumina refineries. In recent ages it has found many applications towards Advanced Carbon Nano Tubes, Carbon Electrode, Carbon Fibers ... etc. Being a chief source of carbon, almost all applications of coal want it to be in purest form, thus it needs to be cleaned from any sort of undesired impurities associated with it. ^{[2][3][4]}

1.2. COAL CLEANING ^{[3][4][6]}:

The components of coal which comprise of Shale, Clay, Sandstone, Silica, Pyrite, Gypsum, Sulphates and Phosphates constitute the mineral impurity in coal. Impurities can be of two types: a) Inherent or Fixed and b) Extraneous or Free. The first category gets associated to coal during its formation period while the second one gets associated during the mining activities, storage and transportation, after the coal is formed. Hence, Coal Cleaning, as the name suggests, is the method implemented for the removal/reduction of mineral impurities from the coal and for better and more efficient usage.

The inherent or fixed impurities constitute very negligible part and are also very difficult to clean unless some special techniques like acid or alkali leaching are employed. Thus we always concentrate on the cleaning of Free or Extraneous Impurities.

Broad classification of coal cleaning processes can be seen as in Figure 1.

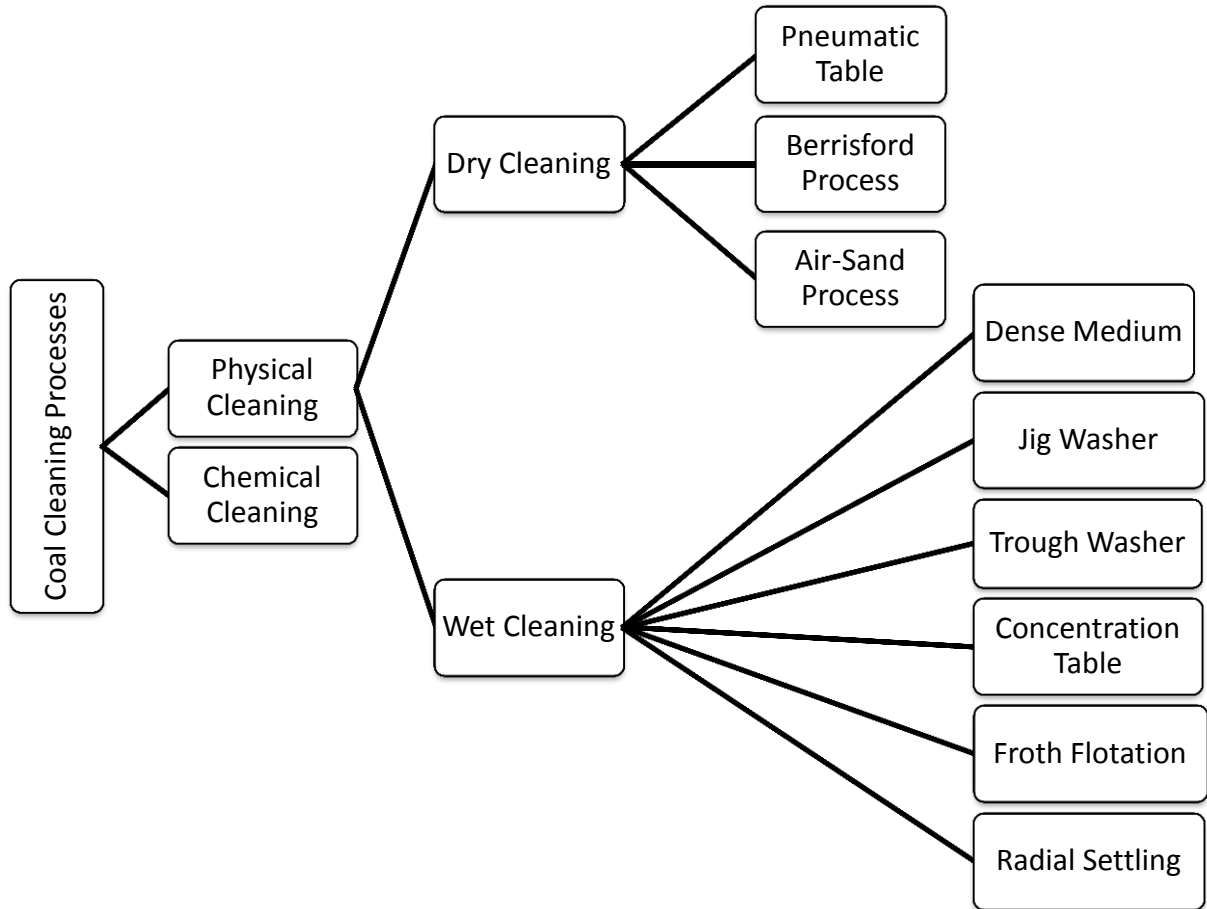


Fig. 1: Classification of Coal Cleaning Processes

Referring Fig. 1, chemical method of cleaning is restricted to laboratory testing. It is also very costly and less efficient. The physical cleaning processes help us cleaning coal on the basis of the differences in the physical properties of pure coal and impurities. Wet and Dry methods can be defined, as predictable, by the type of operation i.e. wet and dry respectively. Majority of dry processes operate on the basis of density, friction, resilience; while most of the wet processes operate on the basis of density, shape, size and wettability.

1.3 ADVANTAGES OF COAL CLEANING ^[6]:

The benefits of coal cleaning can be listed as below:

- i. Cleaned coal is more uniform in size, composition, calorific value and moisture content.
- ii. By reducing the ash or impurity content, coal cleaning contributes to reduced slagging and fouling in furnaces, thus increasing boiler on-stream availability, decreasing maintenance and lowering overall operating cost.
- iii. The transportation and storage cost is reduced. It also leads to less ash disposal problem.
- iv. Due to lowered Sulphur content, clean coal is greener towards the environment by preventing possibility of acid precipitation and other environmental hazards. It also eliminates the setting up of Flue Gas Desulphurization units.
- v. Coal cleaning can be used to tailor the coal to customer specification, thus creating a higher value of the product.
- vi. Sometimes the caking properties of coal improve due to enrichment of vitrain.

1.4 CURRENT STUDY:

Out of all the wet methods of coal cleaning, dense media separation and jigging are predominantly used in commercial practice. While the froth flotation method is not followed on large scale anywhere in the country, this opens avenues for a worthy area of experiments and research. The above listed two methods cannot clean fine coal below 1 mm while froth flotation can. Moreover the demand of ultrapure fine coal is increasing to meet the need of industries such as carbon electrode, carbon fibre, carbon nanotubes ... etc. Hence, we wish decided to carry out the studies on froth flotation technique applied to field of coal cleaning.

CHAPTER – 2

LITERATURE SURVEY

2. LITERATURE REVIEW

2.1 FROTH FLOTATION:

Froth flotation can be coined as a highly adaptable method for the physical separation of particles based on the differences in the ability of air bubbles to selectively adhere to the specific mineral surfaces in the mineral or coal slurries. When attached to air bubbles the particles are carried to the surface and removed gradually, while the particles which are completely wetted stay in the liquid phase. Froth flotation can be implemented to a wide range of mineral separations as it uses chemical treatment methods to selectively modify mineral surfaces so that they possess the necessary properties for the separation. Currently it is being used as a versatile method in separating sulphide minerals from silica gangue (and from the other sulphide minerals); separating potassium chloride from sodium chloride; separating coal separating coal from ash-forming minerals; removing silicate minerals from iron ores; separating phosphate minerals from silicates; and even non-mineral applications such as de-inking recycled newsprint. Whether it is fine-grained ores or coals that are not suitable for gravity concentration, it is particularly useful ^[8].

The advantages are:

- i. It is suitable for fine coals (< 1 mm) e.g. mine dust, slack coal, washing rejects, slurries ... etc.
- ii. It is especially used for producing ultra-pure coal for electrode carbon manufacture.

The disadvantage can be its high capital and running cost. Moreover, it cannot clean lumpy coals unless crushed to powder form.

2.2 RECENT TRENDS AND DEVELOPMENTS:

Froth flotation operation is a highly complicated phenomenon as there are three phases involved in this i.e. solid coal or mineral, water as slurry medium and air bubbles which in turn makes it a challenging area of study. Despite of being introduced in 1900s and after numerous years of research and development this method of cleaning remains relatively less efficient and is not fully understood. Though widely used for the cleaning of mineral ores, it is still not efficiently used for coal cleaning purposes.

According to the generalization done by *Prof Surendra K. Kawatra, Department of Chemical Engineering, Michigan Technological University*, the phenomenon of froth flotation depends on the following three systems being specific ^[8]:

i. Equipment Components:

Cell design, Agitation, Air flow, Cell bank configuration, Cell Bank control

ii. Operation Components:

Feed rate, Mineralogy, Slurry density, Particle size, Temperature

iii. Chemical Components:

Frothers, Collectors, Activators, Depressants, pH controller

Thus the area of research related to Froth Flotation revolves around the study of the effect of such components on the efficiency of cleaning using froth flotation the aforesaid technique.

According to *Laurila et al. (2002)*, the major variables in this process can be listed as [10]:

- Slurry flow rate (or retention time)
- Slurry composition (Nature of solid content, concentration)
- Chemical reagents (Frothers, collectors, pH controllers)
- Electrochemical potential (Eh) and Conductivity
- Froth properties (Bubble size, density, froth stability)
- Slurry levels and air flow rates

If we wish to change all the parameters simultaneously we cannot achieve satisfactory cleaning. Thus we always try varying one variable at a time keeping all the other parameters intact. Moreover, some of these are associated strictly to mineral beneficiation; thus have less importance in the study of fine coal cleaning.

Some of the notable research results can be summarized as below:

- 1) Experiments show that use of classic oils and lubricating oils can improve the performance in froth flotation. Without using any collecting agent, Ukraine coal was tested to give very low wt. % of coal with lowest ash content. But high combustible recoveries had been obtained using fuel-oil and kerosene while the ash rejection was low. On an overall basis bright stock and diesel oil were observed to produce best result for the same coal sample. It was also inferred that the type of oil used may partially depend on the type and origin of coal subjected to froth flotation. ^[11]

- 2) Chemical reagents and their mixtures are sometime very much effective in enhancing the froth flotation and thus are important factors. Parameters such as reagent addition strategy, nature and particle size of the reagent, amount of reagent, level of adsorption of reagents on solid surfaces are decisive in efficiency of coal cleaning. According to tests run on bituminous coal using non-ionic reagents such as Triton-x-100, Brij-35, MIBC (Methyl isobutyl carbinol), SDS (Sodium dodecyl sulphate), the ash rejection and combustible recovery was enormously enhanced. ^{[12][14]}

- 3) When Ultrafine particles were observed to show negative effect on trends of froth flotation. Stability and mobility of froth become important for low rank coals. When components such as fluorapatite and other minerals are present in the coal sample, it can cause problems in combustion by generating corrosive vapors. Attachment of fine coal particles to pulp surface and bubble stability are improved by adding kersone. ^[14]

- 4) Instrumentation and process control have also been major area of research. Following controls are attempted such as ^{[9][10]}
 - a. Instrumentation control: Valves, measurement sensors, XRF...
 - b. Base level control: Slurry rates, aeration rate, reagent dosing
 - c. Advanced control: Process disturbance, grade and recovery control
 - d. Optimization: Maximum profit

- 5) Emphasis has been given on increasing the efficiency by increasing the size of froth flotation cell, increasing the automation in charging and recycling, uniform air dispersion and bubble size analyzer. Use of software packages such as FloatStar Optimizer,

VisioFroth, Frothmaster and many others has been trialed and successfully implemented in industries. ^[9]

- 6) Detailed study of recent research works suggests that the effect of chemical reagents is studied with more interest in comparison of other variables. Since activators and depressants are usually restricted to mineral beneficiation, frothers and collectors are important when we deal in fine coal cleaning. As recent technologies are being developed, the CFD simulation of flotation chamber and also the digital imaging of froths are being carried out in order to study the frothing and froth stability.

Thus the present project work focuses on the study on effect of key variables on froth flotation and its efficiency in cleaning coals.

CHAPTER – 3

MATERIALS AND METHODS

3. MATERIALS AND METHODS

The following experimental methods are being carried out as a part of this research project.

3.1 SAMPLE COLLECTION AND PREPARATION:

For the experiments coal was collected from Basundhara open cast project (Seam-3). At first lumpy coal was subjected to jaw crusher.

- a. For proximate analysis, coal was then intermixed thoroughly and sampling was done by coning and quartering. This was meant to attain further uniformity in the obtained coal sample. Some amount of coal was kept aside for proximate analysis.
- b. Rest of the coal was subjected to ball mill for crushing to finer size. Small quantity of coarser coal was found; those were screened, crushed again and mixed in the obtained powdered coal.

3.2 PROXIMATE ANALYSIS:

Determination of moisture, volatile matter, ash and fixed carbon in coal comprises its proximate analysis. It suggests us the overall composition of coal without incorporating elemental analysis. It also gives a picture of uniformity in the coal sample.

Determination of Moisture Content:

Approximate 1 g fine coal was taken in a weighed porcelain crucible and was placed in a hot air oven at 100 – 110 degree C for 1 hour. Then the loss in weight of the coal due to this heating gave us the moisture per cent of coal used.

$$\% \text{ Moisture in coal} = \frac{\text{Loss in weight of coal}}{\text{Weight of coal initially taken}} \times 100$$

Determination of Volatile Matter in Coal:

It is the loss in weight of moisture free powdered coal when heated in a crucible fitted with a loosely fitting cover in a muffle furnace at 950 degree C for exactly 7 minutes.

$$\% \text{ Volatile matter in coal} = \frac{\text{Loss in weight of moisture free coal}}{\text{Weight of moisture free coal}} \times 100$$

Determination of Ash in Coal:

It is the weight of residue left in a crucible after complete combustion of a previously weighed quantity of powdered coal in an open crucible (i.e. in the presence of air) at 750 degree C in a muffle furnace for duration of 90 minutes.

$$\% \text{ Ash in coal} = \frac{\text{Weight of residue ash formed}}{\text{Weight of coal initially taken}} \times 100$$

Determination of Fixed Carbon:

It was mathematically calculated and was determined indirectly by deducting the sum of total of moisture, volatile matter and ash percentage from 100.

$$\% \text{ Fixed carbon in coal} = 100 - (\text{moisture \%} + \text{volatile matter \%} + \text{ash \%})$$

The proximate analysis was carried out for 3 specimens from the same sample to check the correctness and to ensure uniform result throughout. It was reported in tabulated manner and average value of Ash % and Fixed Carbon % were obtained.

3.3 WASHABILITY STUDIES:

Cleaning or washing process generally depends upon the differences in density between coal particles and its impurities. The extent of removal of free dirt or the amenability of a coal to improvement in quality is more commonly known as the ‘Washability’ of coal and is usually carried out by Float and Sink test. This Washability study helps us in design of washeries and coal processing plants, in techno-economic evaluation and day-to-day plant control.

3.3.1 Float and Sink Test:

The crushed coal sample was sieved and size fraction of $-2 \text{ mm} + 1 \text{ mm}$ was obtained. The organic liquids used in this method were Carbon Tetrachloride (sp. Gravity 1.595, Benzene (sp. Gravity 0.878) and Bromoform (sp. Gravity 2.889). By inter-mixing these liquids, liquids of specific gravities 1.3, 1.4, 1.5, 1.6, 1.7, 1.8 were prepared. The component calculation is given in Table 3. Due to limited availability of organic liquids, the test was carried out in small scale in 250 mL beakers. The beakers were arranged in the increasing order of their specific gravity. The specific gravity was measured using Hydrometer; when any deviation was found w.r.t. the desired specific gravity, further organic liquids were added to achieve correct specific gravity.



Fig. 2: Float and Sink Test

50 g sample was first placed in the lowest specific gravity fluid i.e. 1.30. The fraction lighter than the liquid did float and the heavier fractions did sink. The sink was then dried and placed in the next heavier liquid and as earlier, the float and sink fractions were separated, and the sink was again put into next higher density liquid, it was carried out up to the 1.80 fraction. In this way the fractions from different densities were collected, dried and weighed. The ash analysis of all coal was done and reported.

3.3.2 Washability Curve:

With detailed calculations for total float-ash %, total sink-ash % and cumulative yield up to middle fractions w.r.t. yield %, washability curves were drawn and reported.

3.4 FROTH FLOTATION CELL:

After the complete washability studies had been done, the froth flotation tests were carried out in the froth flotation cell at the Material Handling Laboratory. Volume of the cell is 9 L and this is usually operated with this amount of slurry. The schematic diagram and real photograph of the froth flotation cell used in the experiments are given below.

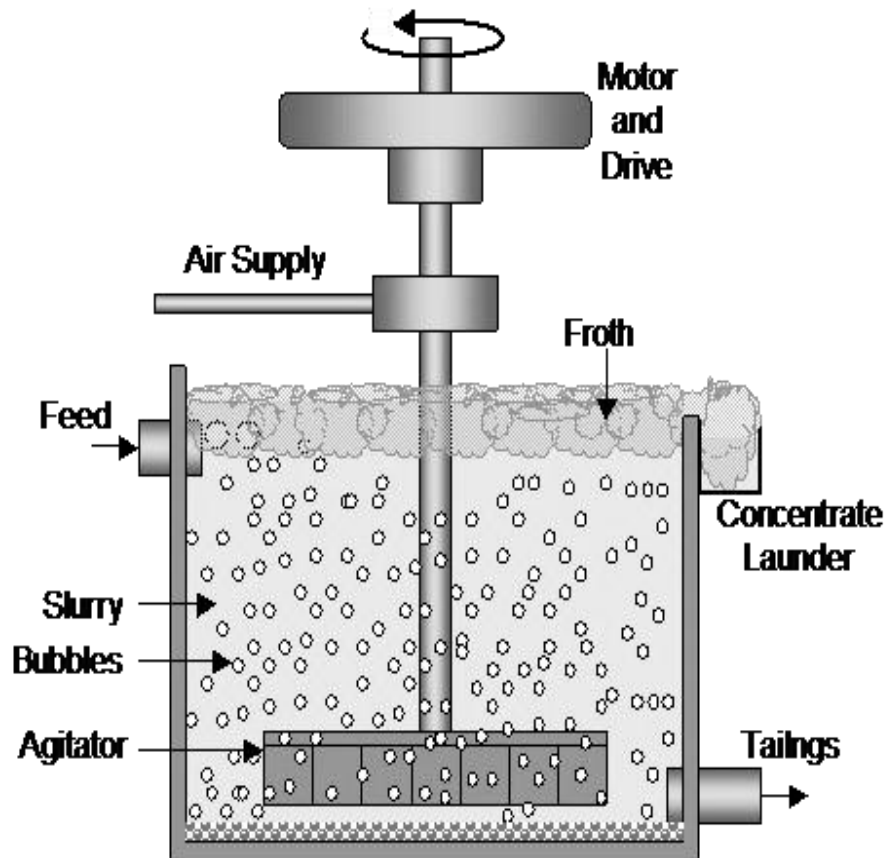


Fig. 3: Schematic diagram of a froth flotation cell ^[17]



Fig. 4: Photograph of Froth Flotation Unit in Laboratory

From the Fig. 4 we can see that the set-up is supplied with a motor drive along with an axial agitator for agitation. Feed is supplied at known composition and air is supplied to the mixture. Upon agitating this mixture, the presence of Frothers enables bubble or froth formation. The subsequent froth can be collected as overflow and the tailings are collected as rejects from the cell. The froth is dried and analysed for weight of ash content per cent. Similarly the rejected tailings are also analysed.

3.5 FROTH FLOTATION TESTS:

3.5.1 Constraints:

Froth flotation cell used in the laboratory operates in a semi-batch mode i.e. the slurry is fed to the system once and the froth is collected continuously while the tailings are collected at the end of the experiment.

Being a single semi-batch cell many options of control are directly eliminated; for this cell the parameters that cannot be varied are:

- a. Volume of the cell
- b. Slurry flow rate
- c. Air flow rate (aeration)
- d. Rate of recycling
- e. Speed of agitator
- f. Retention Time

3.5.2 Parameters in Current Study:

With the above said limitations, we were left with following parameters for study:

- a. Slurry concentration
- b. Coal particle size
- c. Collecting agent
- d. Frothing agent
- e. pH of Separating medium

3.5.3 Effect of Slurry Concentration:

To study the effect of coal slurry concentration, it was desired to prepare slurries of different concentrations. At first the fine coal particles obtained from the ball mill were subjected to screening using a BSS 72 mesh screen ($-252\ \mu\text{m}$). In order to get concentrations of 25 g/L, 50g/L, 75 g/L and 100 g/L in 9 L of cell volume 225 g, 450 g, 675 g and 900 g of coal powder was weighed and kept separately.

Then in 9 L of tap water 225 g of coal was thoroughly mixed and the slurry was fed to the froth flotation cell. With the air inlet closed, the slurry was at first agitated for few minutes to obtain more uniform slurry. Sooner 4-5 drops of Pine oil and Oleic acid were added to the cell as frothing agent and collecting agent respectively. As soon as the froth formation started, all the froth was collected continuously in a bucket. After duration of 10 minutes froth formation was about to cease and 10 minute was regarded as the standard time for further runs of froth flotation. At the end tailings tap was opened and collected in a small amount in a conical flask.

All the froth collected was placed on large filter paper with the help of funnel and conical flask. Collected tailing was subjected to filtration. Both the froth and tailings were left for air drying under normal atmosphere for 4-5 days.

Similar to the above said procedure, other concentrations 50g/L, 75 g/L and 100 g/L were prepared, tested and the ash % was obtained. Mathematically % AR, % CR and EI were calculated and plotted against corresponding concentrations of coal slurry (C, g/L).



Fig. 5: Image of froth at the onset of run ($t=0$ min)

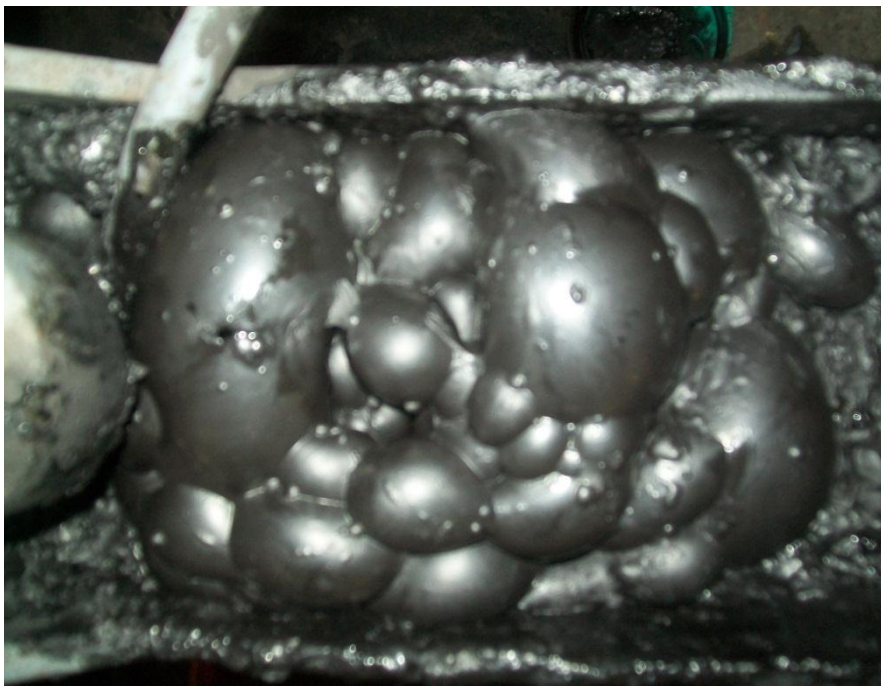


Fig. 6: Image of froth at the end of run ($t=10$ min)

3.5.4 Effect of Particle Size in Slurry:

In order to study the effect of coal particle size in the feed slurry, it was desired to prepare slurry of a specific concentration using different coal size. We fixed the concentration to be 50 g/L and size fractions of -150 μm , -180 μm , -212 μm , -300 μm and -425 μm were obtained using corresponding BSS mesh screens.

Though the theory of froth flotation says that it is suitable for particle size < 1 mm but recent studies indicate that the efficiency decreases considerably above -500 μm . In these sets of experiments, the data of -212 μm was taken from the previous set of experiments of slurry concentration. Moreover due to non-availability of advanced pulverizer, we could not find considerable amount of coal fractions below 150 μm .

Coal slurry of 50 g/L concentration was prepared by mixing 450 g of coal of -150 μm size in 9 L water. In similar to above methods, slurry was fed and froth flotation was carried out for 10 minutes. Froths and tailings were collected carefully and left for drying which were then analysed for % ash content. This was carried out for rest of the size fractions and the corresponding observations were reported. % AR, % CR and EI were calculated from all these observations and plotted against corresponding particle sizes in coal slurry (X, μm).

3.5.5 Effect of Collecting Agents and Doses:

Referring to studies on *Ukraine coal by Ibrahim Sonmez and Yakup Cebeki, Cumhuriyet University*, it is concluded that classic oils such as Diesel, Kerosene and lubricants (bright stock, spindle oil) were very much effective in enhancing the efficiency of coal cleaning. As stated earlier the collecting agent used in the previous runs was Oleic acid which has been

called obsolete in current research scenario; thus better alternatives of collecting agent were suggested as Kerosene, Diesel, Lubricant and their mixture.

Three collecting agents were planned to study i.e. pure kerosene, pure diesel and a mixture of kerosene, diesel and Servo 2T lubricant (in the vol. ratio 2:1:2).

Keeping the slurry concentration to be 50 g/L, particle size to be -252 μm and frothing agent to be Pine oil, froth flotation runs were done using different collecting agents at different doses (i.e. concentration). 1 ml, 2.5 ml, 5 ml and 10 ml doses were fed to the cell and the corresponding weights of doses were calculated using their specific gravities. % AR, % CR and EI were calculated and plotted against corresponding type of collecting agents and their respective doses (D, g/kg of feed coal)

3.5.6 Calculations and Graphical Interpretation:

The analysis of all the cleaned coal was carried out in terms the ‘% Combustible Recovery (CR)’ and ‘% Ash Rejection (AR)’ calculations. The mathematical formulae are given below. For this the weight of recovered coal and its ash content was also to be determined prior to the calculations.

$$\% \text{ Ash Rejection} = \left(1 - \frac{W_c A_c}{W_f A_f} \right) \times 100$$

Where, A_c = Ash content of clean coal

A_f = Ash content of feed

W_c = Mass of clean coal

W_f = Mass of feed

$$\% \text{ Combustible Recovery} = \left[\frac{W_{c,daf}}{W_{f,daf}} \right] \times 100$$

Where, $W_{c,daf}$ = Mass of dry-ash free clean coal $W_{f,daf}$ = Mass of dry-ash free feed

For % CR,

$$W_{f,daf} = W_f(100 - (M + Af))/100$$

$$W_{c,daf} = W_c(100 - (M + Ac))/100$$

Where, M is the average moisture amount of coal, taken as 5 %.

Another quantitative figure ‘Efficiency Index of Flotation Cell’ was also useful in determining the effect of variables and the efficiencies. Mathematically it is given by

$$\text{Efficiency Index} = (\% AR + \% CR) - 100$$

As mentioned earlier, the effect of key variables were studied by plotting the % AR, % CR and Efficiency Index against the varying the parameters and then discussed further.

Weight of coal in the tailings, W_r was also calculated from

$$W_r = W_f - W_c$$

CHAPTER – 4

RESULTS AND DISCUSSION

4. RESULTS AND DISCUSSION

4.1. PROXIMATE ANALYSIS:

The observations and calculations of proximate analysis are reported below:

Table 1: Observations of Proximate Analysis:

Sample No.	Weight of Empty Crucible (g)	Weight of Sample + Crucible (g)	Weight of coal content (g)	Weight after moisture removal (g)	Weight after Volatile matter removal (g)	Weight after ash residue formation (g)
1	21.43	22.43	1.00	22.38	22.06	21.72
2	22.57	23.57	1.00	23.52	23.19	22.86
3	21.07	22.07	1.00	22.02	21.70	21.34

Table 2: Results of Proximate Analysis:

Sample No.	Moisture (M) %	Volatile Matter (VM) %	Ash (A) %	Fixed Carbon (FC) %
1	5.00	33.68	29.00	32.32
2	5.00	34.74	29.00	31.26
3	5.00	33.68	27.00	34.32

The average ash % is 28.33 % and average fixed carbon % is 32.63 %.

4.2.WASHABILITY STUDIES:

Table 3: Composition of Separating Medium (of 200 mL) for Float and Sink Test:

Specific gravity	CCl ₄ (ml) [Specific gravity- 1.595]	Benzene (ml) [Specific gravity- 0.878]	Bromoform (ml) [Specific gravity- 2.889]
1.3	117.70	82.30	-
1.4	145.60	54.40	-
1.5	173.50	26.50	-
1.6	197.90	-	2.10
1.7	182.60	-	17.40
1.8	167.20	-	32.80

Table 4: Washability Data:

Specific Gravity	Weight of Each fraction (Float) g.	Ash of each fraction (Float) %	Yield of total Float %	Cumulative Yield of Float %	Ash of Total Float %	Yield of Total Sink %	Ash of Total Sink %	Cumulative Yield up to Middle Fraction %
	W	A	C	D	E	F	G	H
1.30	1.06	6.25	2.13	2.13	6.25	97.88	28.81	1.06
1.40	5.18	10.12	10.36	12.49	9.46	87.51	31.02	7.31
1.50	8.07	16.98	16.14	28.63	13.70	71.37	34.20	20.56
1.60	14.36	22.35	28.73	57.35	18.03	42.65	42.18	42.99
1.70	11.17	32.63	22.34	79.69	22.12	20.31	52.68	68.52
1.80	6.94	39.85	13.88	93.57	24.75	6.43	80.37	86.63
> 1.8	3.22	48.92	6.43	-	26.31	-	-	96.79

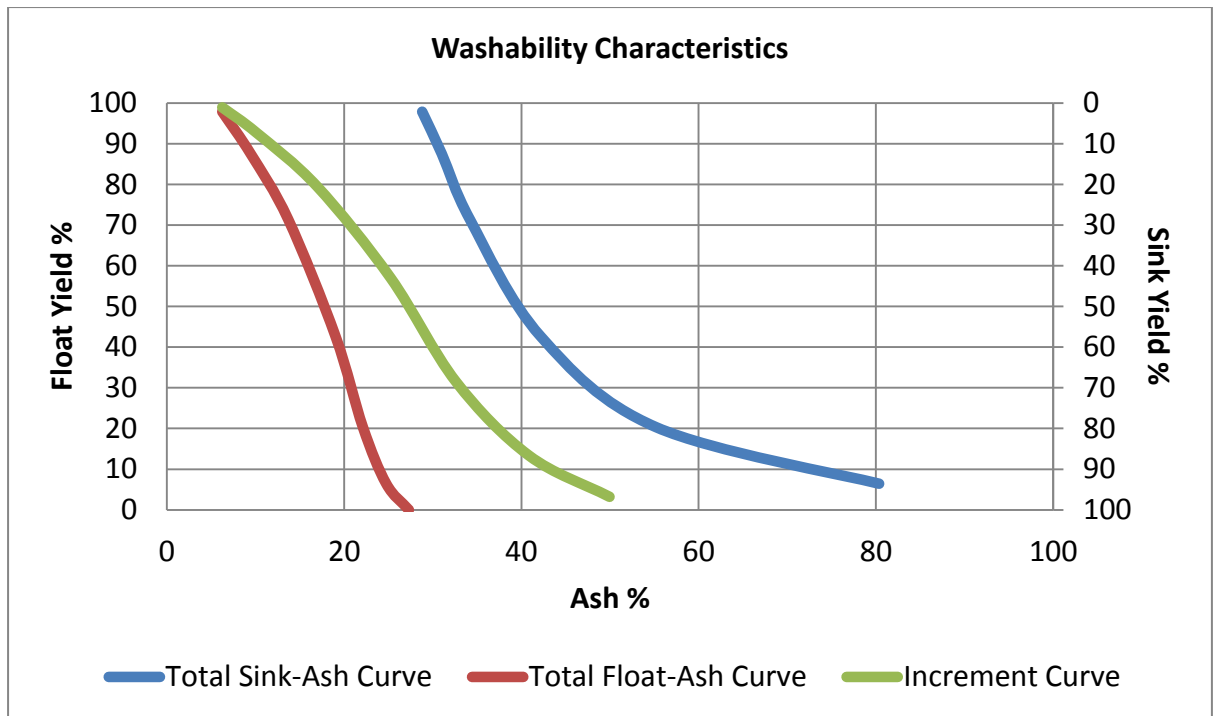


Fig. 7: Washability Characteristics Curve

4.3.EFFECT OF CONCENTRATION OF COAL SLURRY:

Table 5: Data of Clean Coal and Tailings-1:

Sl. No.	Concentration of Coal Slurry (g/L)	Weight of Clean Coal (g)	% Ash in Clean Coal	% Ash in Tailings
1	25	52	14	35
2	50	66.12	15	33
3	75	78.27	17	32
4	100	95.67	18	30

Table 6: Calculation for Effect of Concentration of Coal Slurry:

Sl. No	C (g/L)	W _f (g)	W _c (g)	W _r (g)	A _f	A _c	% AR	W _{f,daf} (g)	W _{c,daf} (g)	% CR	EI
1	25	225	52.00	173.00	28.33	14.00	88.58	150.01	42.12	28.08	16.66
2	50	450	66.12	383.88	28.33	15.00	92.22	300.02	52.90	17.63	9.85
3	75	675	78.27	596.73	28.33	17.00	93.04	450.02	61.05	13.57	6.61
4	100	900	95.67	804.33	28.33	18.00	93.25	600.03	73.67	12.28	5.52

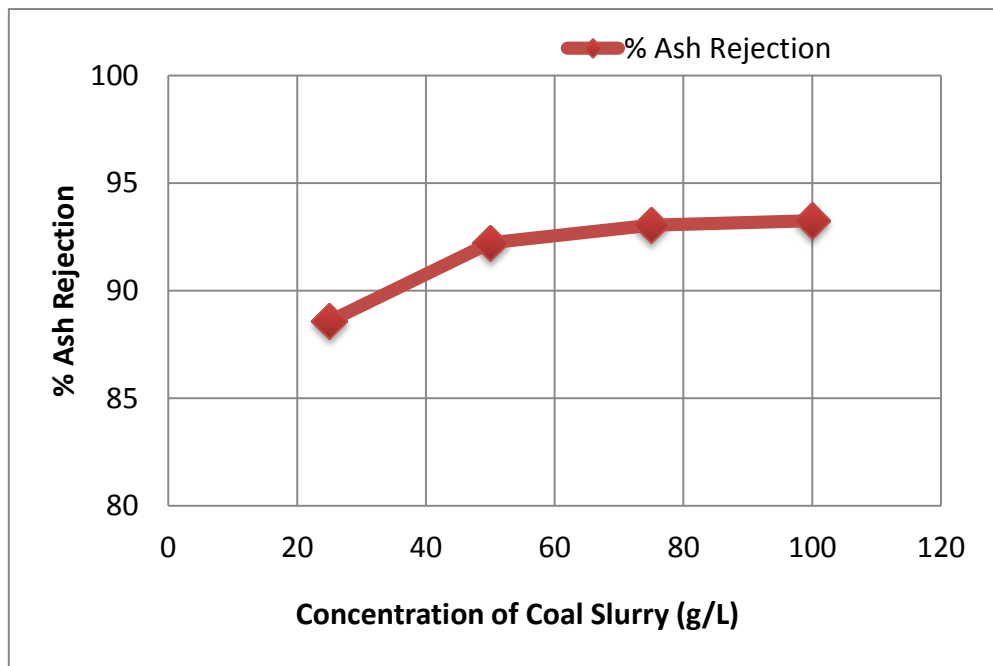


Fig 8: % Ash Rejection vs Concentration of Coal Slurry

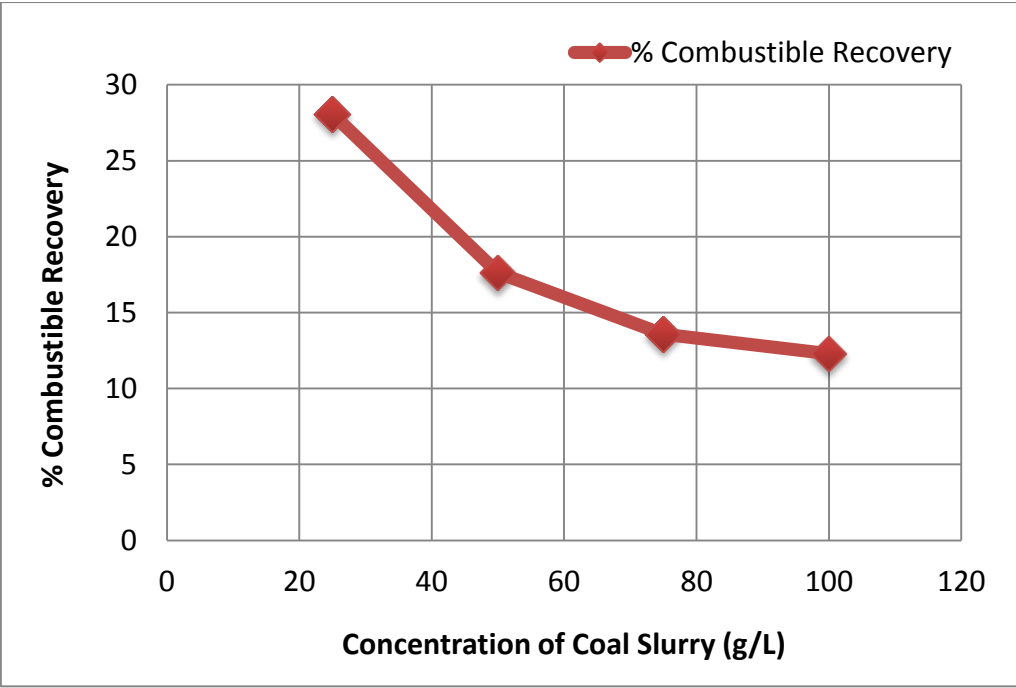


Fig 9: % Combustible Recovery vs Concentration of Coal Slurry

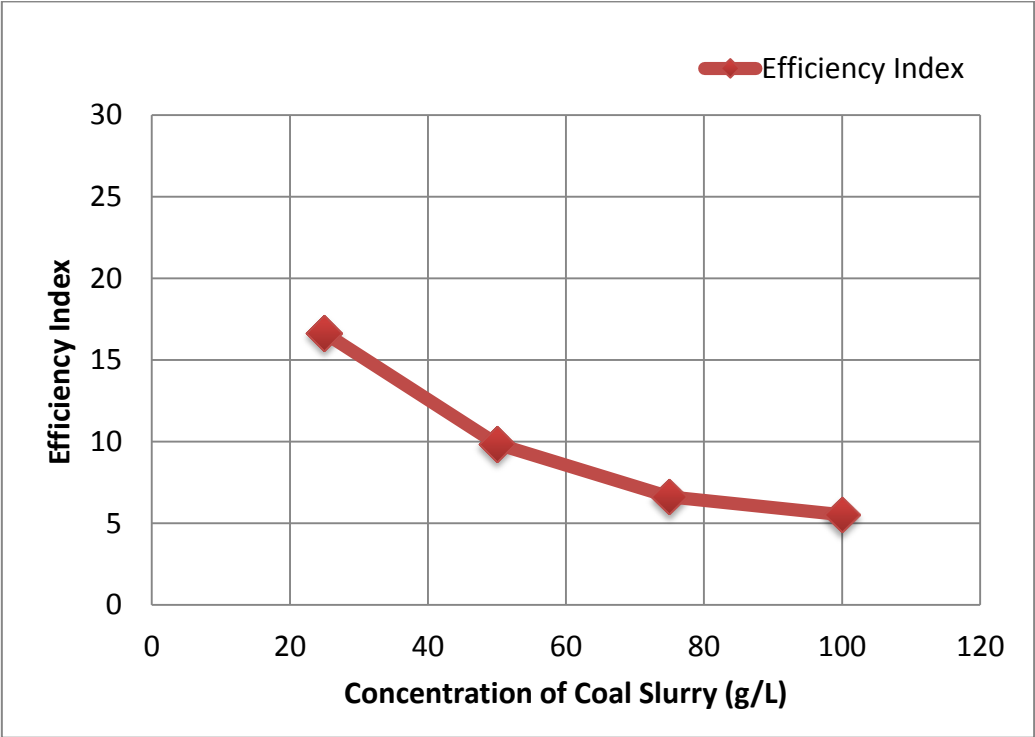


Fig 10: Efficiency Index vs Concentration of Coal Slurry

4.4.EFFECT OF PARTICLE SIZE IN SLURRY:

Table 7: Data of Clean Coal and Tailing-2:

Sl. No.	Size Fraction (μm)	Weight of Clean Coal (g)	% Ash in Clean Coal	% Ash in Tailings
1	150	70.22	15.00	30.00
2	180	66.00	14.00	30.00
3	212	66.12	15.00	30.00
4	300	63.98	16.00	29.00
5	450	62.37	18.00	30.00

Table 8: Calculation for Effect of Particle Size in Coal Slurry:

Sl. No	X (μm)	W_f (g)	W_c (g)	W_r (g)	A_f	A_c	% AR	$W_{f,daf}$ (g)	$W_{c,daf}$ (g)	% CR	EI
1	150	450	70.22	379.78	28.33	15.00	91.74	300.02	56.18	18.72	10.46
2	180	450	66.00	384.00	28.33	14.00	92.75	300.02	53.46	17.82	10.57
3	212	450	66.12	383.88	28.33	15.00	92.22	300.02	52.90	17.63	9.85
4	300	450	63.98	386.02	28.33	16.00	91.97	300.02	50.54	16.85	8.82
5	425	450	62.37	387.63	28.33	18.00	91.19	300.02	48.02	16.01	7.20

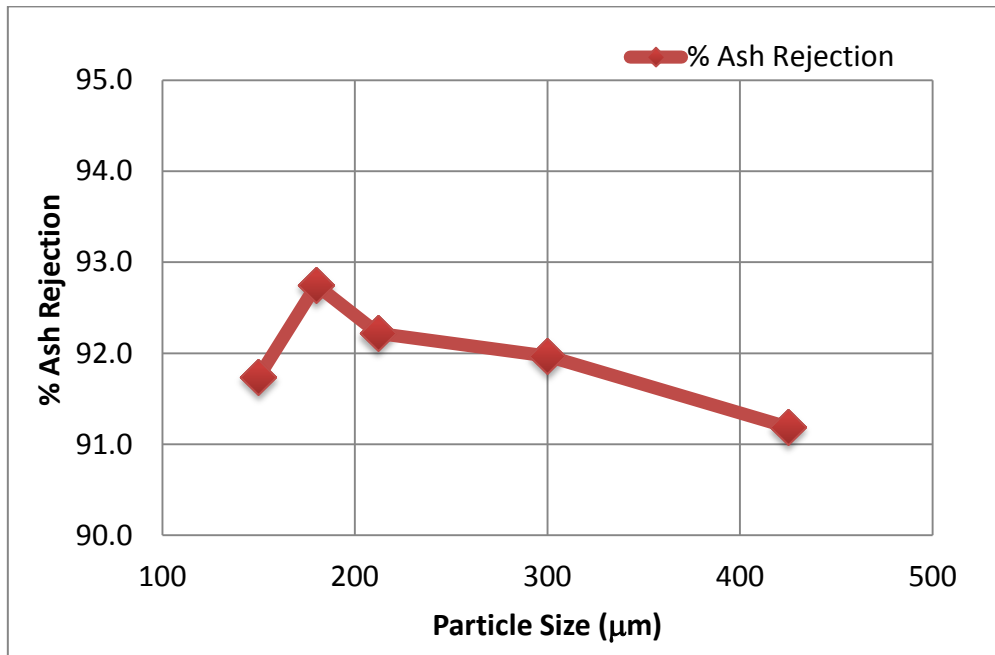


Fig 11: % Ash Rejection vs Particle Size in Slurry

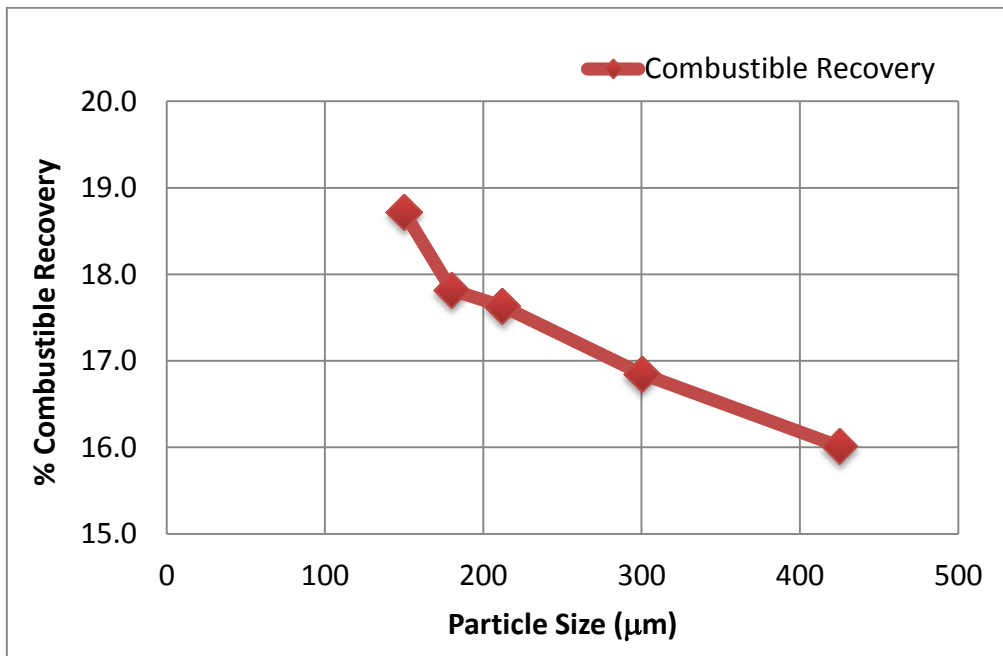


Fig 12: % Combustible Recovery vs Particle Size in Slurry

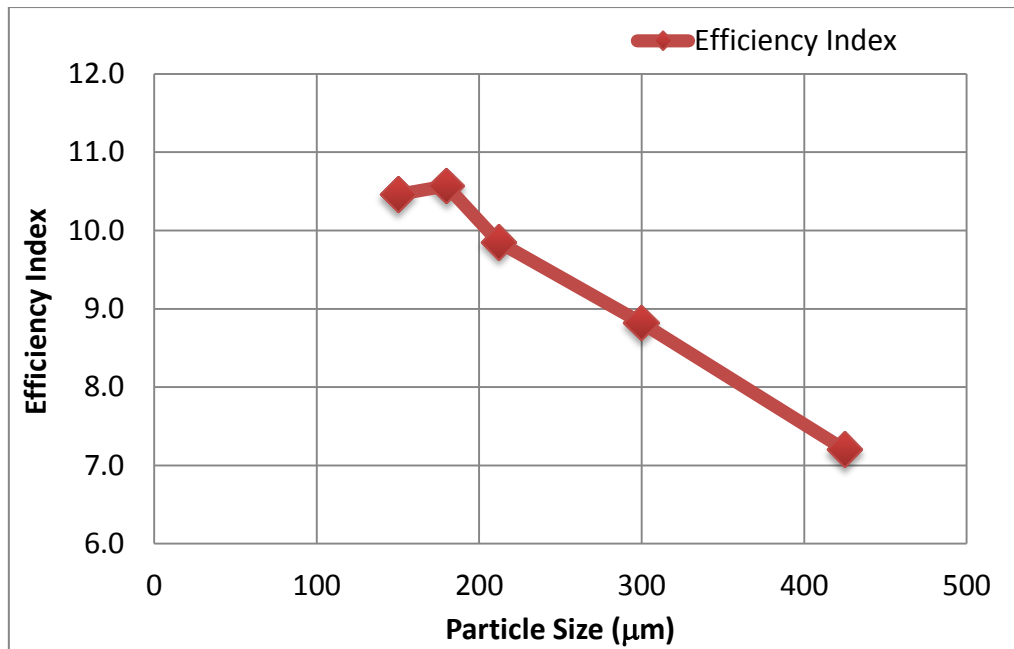


Fig 13: Efficiency Index vs Particle Size in Slurry

4.5.EFFECT COLLECTING AGENTS AND DOSES:

Density of Mixture of oil: 0.780 g/ml; Kerosene: 0.766 g/ml; Diesel: 0.756 g/ml

Table 9: Data of Clean Coal and Tailing-3: [using Mixture of Oils]

Sl. No.	Dose (ml)	Dose (g/run)	Dose (g/kg of feed)	Weight of Clean Coal (g)	% Ash in Clean Coal	% Ash in Tailings
1	0.000	0.000	0.000	60.530	21.000	30.00
2	1.000	0.780	1.733	68.920	17.000	30.00
3	2.500	1.950	4.333	79.520	14.000	31.00
4	5.000	3.900	8.667	91.050	11.000	33.00
5	10.000	7.800	17.333	95.670	10.000	33.00

Table 10: Data of Clean Coal and Tailing-4 [using Kerosene]:

Sl. No.	Dose (ml)	Dose (g/run)	Dose (g/kg of feed)	Weight of Clean Coal (g)	% Ash in Clean Coal	% Ash in Tailings
1	0.00	0.000	0.000	60.53	21.00	30.00
2	1.00	0.766	1.702	67.82	17.00	34.00
3	2.50	1.915	4.256	78.00	14.00	32.00
4	5.00	3.830	8.511	90.00	11.00	31.00
5	10.00	7.660	17.022	93.52	10.00	30.00

Table 11: Data of Clean Coal and Tailing-5: [using Diesel]

Sl. No.	Dose (ml)	Dose (g/run)	Dose (g/kg of feed)	Weight of Clean Coal (g)	% Ash in Clean Coal	% Ash in Tailings
1	0.00	0.000	0.000	60.53	21.00	30.00
2	1.00	0.756	1.680	72.76	16.00	30.00
3	2.50	1.890	4.200	78.92	14.00	32.00
4	5.00	3.780	8.400	90.47	11.00	32.00
5	10.00	7.560	16.800	86.11	11.00	31.00

Table 12: Calculations for the effect of Collecting agents and their doses:

Agent	Sl. No	D (g/kg of feed)	W _f (g)	W _c (g)	W _r (g)	A _f	A _c	% AR	W _{f,daf} (g)	W _{c,daf} (g)	% CR	EI
Nil	1	0.000	450	60.530	389.47	28.33	21.00	90.03	300.02	44.79	14.93	4.96
Mixture of Oils	2	1.733	450	68.920	381.08	28.33	17.00	90.81	300.02	53.76	17.92	8.73
	3	4.333	450	79.520	370.48	28.33	14.00	91.27	300.02	64.41	21.47	12.74
	4	8.667	450	91.050	358.95	28.33	11.00	92.14	300.02	76.48	25.49	17.64
	5	17.333	450	95.670	354.33	28.33	10.00	92.50	300.02	81.32	27.11	19.60
Kerosene	6	1.702	450	67.820	382.18	28.33	17.00	90.96	300.02	52.90	17.63	8.59
	7	4.256	450	78.000	372.00	28.33	14.00	91.43	300.02	63.18	21.06	12.49
	8	8.511	450	90.000	360.00	28.33	11.00	92.23	300.02	75.60	25.20	17.43
	9	17.022	450	93.520	356.48	28.33	10.00	92.66	300.02	79.49	26.50	19.16
Diesel	10	1.680	450	72.760	377.24	28.33	16.00	90.87	300.02	57.48	19.16	10.03
	11	4.200	450	78.920	371.08	28.33	14.00	91.33	300.02	63.93	21.31	12.64
	12	8.400	450	90.470	359.53	28.33	11.00	92.19	300.02	75.99	25.33	17.52
	13	16.800	450	86.110	363.89	28.33	11.00	92.57	300.02	72.33	24.11	16.68

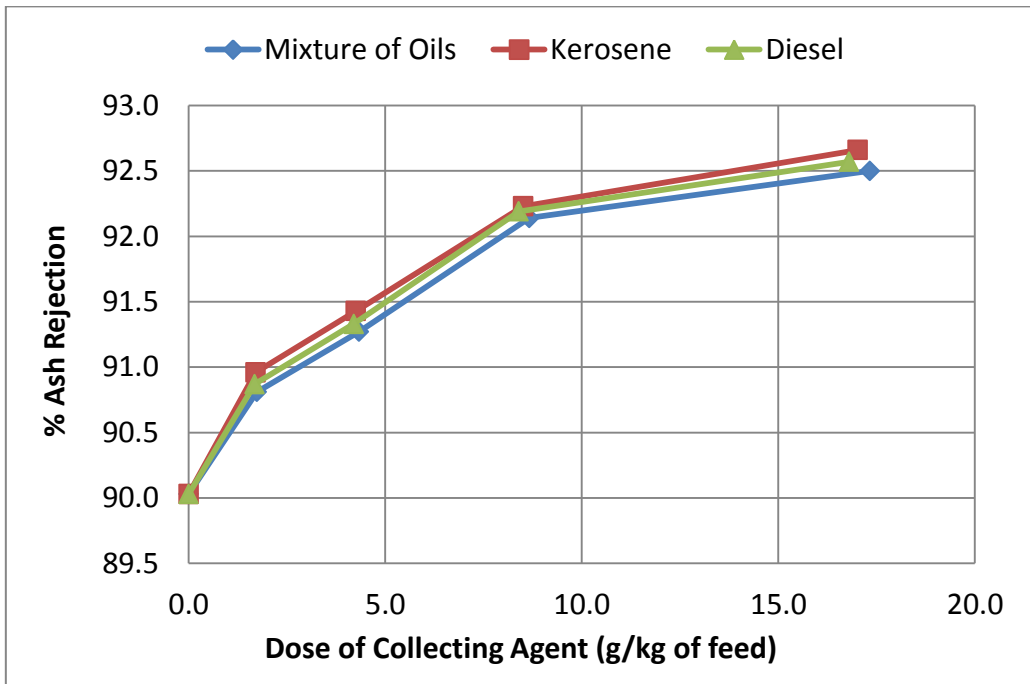


Fig 14: % Ash Rejection vs Collecting agents and doses

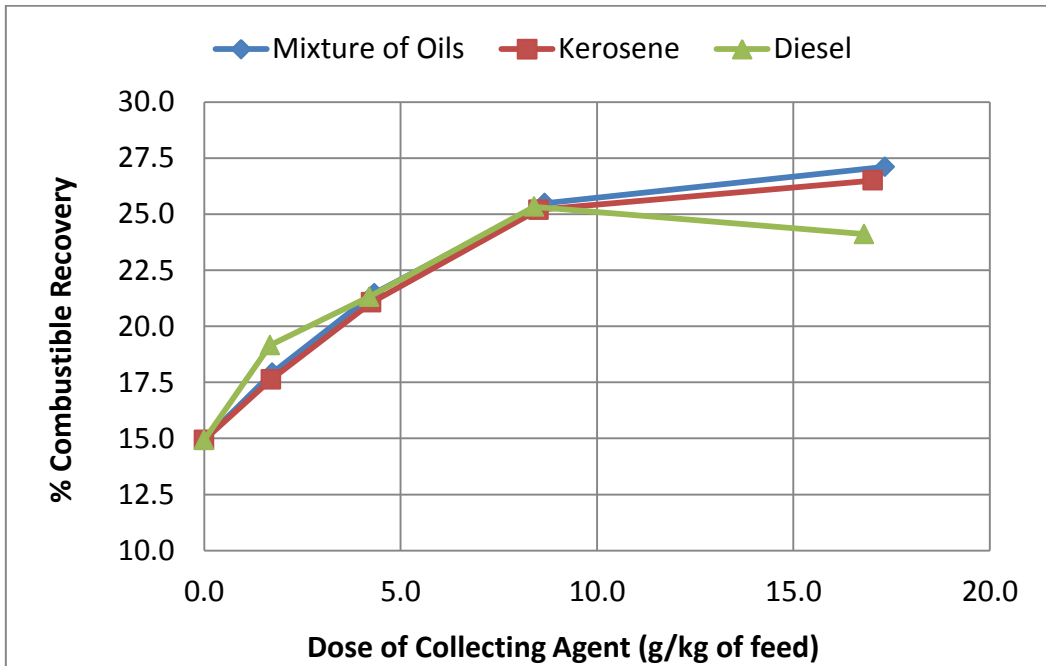


Fig 15: % Combustible Recovery vs Collecting agents and doses

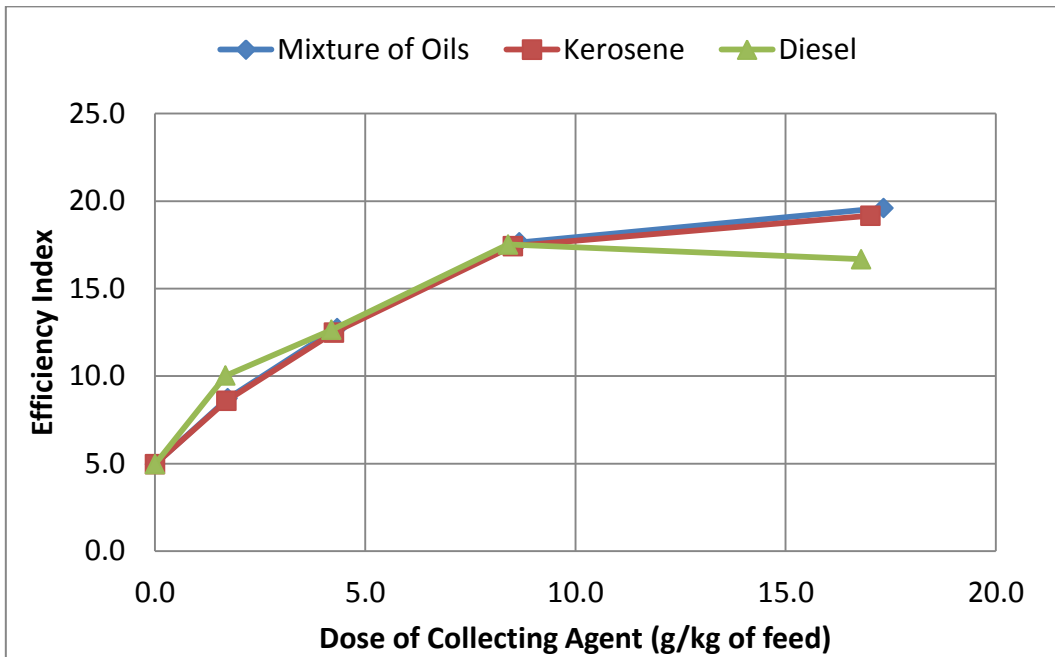


Fig 16: Efficiency Index vs Collecting agents and doses

4.6 DISCUSSIONS:

- The proximate analysis of the obtained coal sample suggested that it is of uniform composition which will prevail for all the runs using the coal of same stock. The % average ash was found to be 28.33 and % fixed carbon to be 32.66.
- While performing float and sink test of the coal sample we could see that the ash at lowest specific gravity (1.3) we obtained coal of 6.25 % ash and at highest specific gravity (>1.8) we obtained coal of 48.92 % ash. The washability characteristics plot does not show any sharp cut on increment curve, which suggests that coal does not have very high washability. From the calculations, we could see that the optimum specific gravity of separation is 1.6 with total float

yield 57.35 at cumulative ash % of 18.03 i.e. on operating the coal in a medium of specific gravity 1.6 we can obtain 57.35 wt. % of coal having 18.03 % ash.

- When we think of coal cleaning using froth flotation, % ash rejection (AR), % combustible recovery (CR) and efficiency index (EI) of flotation cell are the quantitative figures that enable us to know to which extent coal is being cleaned by the said process. While % ash rejection and % combustible recovery also bear qualitative significance, efficiency index is calculated from these quantities only. % Ash rejection shows us to which extent ash is rejected to the tailings and thus higher the %AR, lower is the ash % in the cleaned coal. % Combustible recovery shows the amount of combustible quantities that are recovered from the total amount of combustibles earlier present in the feed itself.

In all the froth flotation processes, it is desired to have both the % AR and % CR as high as possible at the same time. But at times, froth flotation processes lead to results where one of these two is not significantly high; in that case we compromise with the lower value to achieve satisfactory results. This also indicates that a flotation cell at higher efficiency index will clean the coal more efficiently.

- When coal was subjected to froth flotation at different concentration of coal slurries i.e. 25 g/L, 50 g/L, 75 g/L and 100 g/L keeping the reagents and time of operation fixed, we could see that % AR increased with the increase of concentration from 25 to 50 g/L but showed slight increase thereafter. But the % CR decreased considerably with the increase in concentration. The similar trend is shown by the corresponding EI.

The reagent quantities were fixed for higher concentration of coal slurries, which possibly may be a reason behind decrease in % CR and EI.

- Effect of particle sizes in slurry was studied taking size fractions below 150 μm , 180 μm , 212 μm , 300 μm and 425 μm . % AR, % CR and EI show significant increase with decrease of the particle size from 425 μm to 180 μm . But % AR lowered at 150 μm and the value is even less than that of obtained at 212 μm . % CR showed increase 150 μm also but at the same time value of EI dropped at 150 μm .

Theory of froth flotation says that the particle size should be $< 1\text{ mm}$ but in the recent studies fractions used are usually $< 500\ \mu\text{m}$. Reducing the coal particle size improves its surface characteristics and increases its adherence to air bubble by increasing its hydrophobicity. But beyond certain minimum particle size, the physical weight of particles compensates the surface characteristics, for which the dirtier coal particles tend to rise along with bubbles and accumulate in the froth on the surface by increasing the % ash in the concentrate.

Since both the %AR and EI decreased beyond 180 μm , it can be termed as the optimum size of coal particle in the slurry for the operation of the used coal sample in the working flotation cell.

- While studying the effect of collecting agents and their doses on coal cleaning, the densities of collecting agents used did not vary much, thus the doses could be equivalently calculated in terms of g per kg of coal feed.

All the % AR, % CR and EI tend to increase with increase in the dose of collecting agent. % AR value remained almost same for mixture of oils, kerosene and diesel with kerosene showing the maximum value at higher doses. Diesel showed better values of % CR at lower doses while decreased considerably at the maximum dose w.r.t. mixture of oils and kerosene. While at all the dose levels mixture of oils and kerosene showed similar results, at the maximum dose level,

mixture of oils showed better results than kerosene. The similar trend was seen in EI also with increase in dose levels.

- On the contrary to principle of froth flotation, in the absence of collecting agents, the weight of clean coal collected was significantly higher. But it was also observed that the % ash in the cleaned coal was as high as 21 % which suggested that dirtier coal are entrapped among the clean coal particles and was moved to the froth level with the rising bubbles.
- We can clearly see that % AR, % CR and EI values are distinctly better in case of froth flotation run with kerosene, diesel and mixture of oils in comparisons with the previous all runs. Use of oleic acid as collecting agent gave us inferior results, thus use of such classic oils and lubricants proves more useful in achieving better cleaning through froth flotation.
- Studying the overall cleaning by froth flotation with the washability data obtained earlier by float and sink tests, we could achieve coal of minimum 10 %ash with 21.26 % wt. yield. The highest values of EI and % CR were obtained for mixture of oils as collecting agent while highest value of % AR is obtained by kerosene.
- In recent studies on froth flotation, the % AR obtained are as high as 20 to 50 with very high % CR of 40 to 65. The overall efficiency index was found to be in the range of 14 to 35. Clearly the results from this particular froth flotation cell at varying key parameters are inferior to the results obtained from large scale froth flotation cells.

CHAPTER – 5

CONCLUSION

5. CONCLUSION

Froth flotation has been more widely used in mineral ore beneficiation processes than in coal beneficiation. Since we know that the majority of coal cleaning processes are dense medium separations which clearly give better results than froth flotation processes, the scope of froth flotation processes are limited to mine dust, slack coal and washing rejects, slurries. But it eliminates the inability of cleaning fine coals and slurries as in dense medium separations. In recent practices use of advanced frothing agents such as Triton-x-100, Brij-35, MIBC (Methyl isobutyl carbinol), SDS (Sodium dodecyl sulphate) has showed enormously high ash rejection and combustible recovery values. The cost of such reagents is very high in comparison to pine oil, cresol ... etc. thus may not be useful in small scale beneficiation plants that compromise the quality of clean coal to cut the cost. The results obtained in the laboratory are very much different to that of washeries that operate on froth flotation principles on industrial scale because all of them operated in a continuous fashion where feeding and froth collections are continuous. Also the introduction of advanced controllers and software packages such as VisioFroth, FloatStar ...etc. in froth flotation processes are promising in giving better control and better results.

Effect of several frothing agents and their addition rates can be studied as the future scope of this project. Though effect of pH is more prominent in mineral beneficiation systems, it can also be studied to see the impact on coal cleaning by froth flotation. Moreover, all such experiments are needed to be performed on all grades of coal obtained from different origins or seams, to draw more confident conclusions in this direction.

REFERENCES

1. Annual Report 2010-2011, Ministry of Coal, Government of India,
<http://coal.nic.in>
2. Sarkar Samir. Fuels and Combustion, 3e, University Press
3. Gupta O. P. Elements of Fuels, Furnaces & Refractories, 5e, Khanna Publishers
4. Chakraborty M. Coal technology development activities in India, Energy, Vol. 11 (1986), pp. 1231-1237
5. Wills Barry A. and Napier-Munn Tim: Will's Mineral Processing Technology, 7e, Elsevier Science & Technology Books
6. Singh Suman P. N. Coal beneficiation (Chapter 10), Chemical technology division, Oak Ridge National Laboratory, Oak Ridge
7. Gagarin S. G., Gyul'maliev A. M., Tolchenkin Yu. A.: Trends in Coal Beneficiation: A Review, Coke and Chemistry, 2008, Vol. 51, No. 2, pp. 31–42. © Allerton Press, Inc. (2008)
8. Kawatra S. Komar. Froth Flotation Fundamentals. Michigan Technical University.
www.chem.mtu.edu/chem_eng/faculty/kawatra/Flotation_Fundamentals.pdf
9. Shean B. J., Cilliers J. J.: A review of froth flotation control, International Journal of Mineral Processing, Vol. 100 (2011), pp. 57-71
10. Laurila H, Karesvuori J, Tiili O. Strategies for instrumentation and control of flotation circuits, Mineral processing plant design, practice and control. Vol. 1 (2002), 2174-2195.
11. Sonmez Ibrahim, Cebeci Yakup: Performance of classic oils and lubricating oils in froth flotation of Ukraine coal, Fuel 85 (2006), pp. 1866-1870

12. Erol Murat, Colduroglu Cigdem, Zeki Aktas: The effect of reagents and reagent mixtures on froth flotation of coal fines. *International Journal of Mineral Processing*, Vol. 72 (2003), pp. 131-145
13. Banford A. W., Aktas Z. The effect of reagent addition strategy on the performance of coal flotation, *Minerals Engineering*, vol. 17 (2004), pp. 745-760
14. Woodburn E. T., Flynn S. A., Cressey B. A., Cressey G. The effect of froth stability on the beneficiation of low rank coal by flotation, *Powder Technology*, Vol. 40 (1984), pp. 167-177
15. Demirbas Ayhan. Demineralization of coals via Column froth flotation, *Energy Conversion Management*, Vol. 43 (2002), pp. 885-895
16. Speight James G. *Handbook of Coal Analysis*, 2e, Wiley Interscience Publications, 2005
17. http://year7hawkesdale.wikispaces.com/file/view/froth_flotation2.gif/66318086/394x324/froth_flotation2.gif