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Flotation characteristics of high ash oxidised Indian non-coking coal and its effects on cell flotation

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ABSTRACT

It has been observed that freshly-mined coal floats better than coal which has been exposed to the atmospheric oxygen. It is well known that high rank coals are generally more hydrophobic than low rank coal or oxidised coal. Non-coking coal of Talcher coal field, India has very high ash content and is believed to be surface oxidised. This coal contains around 35-50% ash resulting in low gross calorific value. This coal is very difficult to float even at higher concentration of collector due to its oxidised surface and high ash, thus rendering less hydrophobic to attain complete air bubbles attachment. The investigations have been carried out to improve the floatability of this oxidised, high ash non-coking coal by pre-treating it with chemical reagents. Aliphatic alcohols were selected as pretreatment reagent for this type of coal to examine whether floatability of coal increases. The effect of methanol, ethanol, butanol and octanol were evaluated to determine any improvement in terms of yield and ash content. In the present work, the examination of oxidation property of coal by FTIR spectroscopy revealed a notable change in the hydrophobicity index of the coal due to ethanol treatment. Electro-kinetics study of macerals and bulk sample of coal has also been carried out to confirm the oxidation of coal surface.

Coal flotation depends on coal petrographic composition, particle size distribution, density and ash content and also type of flotation reagents. The flotation characteristics of non-coking coal with respect to mineralogical characteristics, flotation reagents and process variables in Denver D-12 sub-aeration flotation cell have been optimised. As coal is under non-polar substances, non-polar collector diesel oil is used to increase the contact angle. Short chain alcohol MIBC and sodium silicate have been used as frother and dispersant respectively. The effect of other process parameters like pH, conditioning time and rpm of impeller have also been studied. Consumption of chemical reagents have been found to be high in the case of non-coking high ash coal in comparison with coking coal due to low hydrophobicity of coal surface, high ash content and fine size of particles.

Keywords: Coal; Flotation; Hydrophilicity index; Point of zero charge; Flotation reagents

1. INTRODUCTION

The majority of coal reserves in India are thick and inferior in quality. Most of thick seams are banded in nature. The coaly matter in these seams is associated with high ash and the quality becomes worse because of these bands. The bands themselves are usually carbonaceous in nature and very rarely consist of pure sandstones (Prasad, 1989). The basic fact relating to this issues is that the Indian coal seams have basically much higher ash content as compared to the coals obtained internationally. The coal seams in India are formed in geological different situation than elsewhere. Therefore, inherent ash content in Indian coal is much higher. The total coal reserves in India is about 192 billion tonnes and 86.4% comes under the category of non-coking coal which contains mostly 40-50% ash. Higher ash percentage obviously is correlated with poor calorific value. However, the advantages of Indian coal are low sulphur, chloride, iron phosphorous in ash , less toxic elements, high ash fusion temp. and refractory nature of ash (Chowdhary, 1995).

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Depending on the different physical and chemical changes happened during the coalification process, the individual coal attains varying degree of hydrophobicity depending on its origin, rank and surface oxidation. The surface of the coal oxidies depending on weathering condition of the nature and also can occur in the short time between mining and utilisation (Gacia *et al.*, 1991). The oxidation of coal surface increases the hydrophilicity character and it affects on coal flotation (Laskowski, 2001). The rate of oxidation of low rank coal is more in comparison with high rank coal.

Many investigators have tried to improve the floatability of oxidised coal in various methods either by dry or wet process i.e., thermally controlled atmosphere (non-oxidising gaseous environment) and dissolving the oxidised layer using various chemicals caustic soda or in benzyl alcohol (Ye *et al.*, 1988; Brown, 1962; Laskowski, 2001).

Coal flotation depends on coal petrographic composition, particle size distribution, density and ash content (Holuszko *et al.*, 1995; Bensley *et al.*, 1985). When ash content increases, the behaviour of flotation of coal changes due to presence of mineral matter in the coal matrix (Bennett *et al.*, 1983). Coal flotation depends not only on the mineral matter content but also on the mineralogical composition of the mineral matter associated with coal. The adsorption of flotation reagents i.e., collector, frother, activator, depressant or modifier to change the hydrophobicity of the solid plays a vital role for selective flotation. Collectors and frothers are the important reagents which primarily control flotation. Activators, depressants and pH regulators are the secondary reagents, in the flotation of coal fines.

As coal is under non-polar substances, non-polar collectors are used to increase the contact angle. In general the petroleum products like kerosene or fuel oil is used as a collector for coal flotation all over the world. This non-polar collector is water insoluble oily hydro carbon. The mode of action of the water insoluble oily collector is quite different in case of water soluble collector. In this case, the collector species can be transported towards mineral particles only in the form of oil droplets which should attach to the coal particles to facilitate the attachment of bubbles to coal particles (Klassen, 1963; Glembotski, 1970).

A frother has a number of functions in flotation- first, it reduces the surface tension of the air-liquid interface in order that a stable bubbles is produced in the system, secondly, it influences the kinetics of bubble-particle adhesion, thirdly, it thins the liquid layer by interacting with collector molecules and finally stabilizes the bubble-particle aggregates (Schulman *et al.*, 1954; Leja, 1956). The most common frothers used in coal flotation are the short-chain alcohols such as MIBC (Methyl Iso-Butyl Carbinol, also called methyl amyl alcohol or 4-methyl 2-pentanol). Another popular commercial frother is pine oil which contains mostly α-terpineol and its isomers. Since pine oil has some collecting properties for coal and is some what adsorbed on the coal surface, the quantity of pine oil required for an easily floatable coal would be somewhat higher than that of MIBC (Aplan, 1976).

Non-electrolytes and salts of inorganic acids are generally used as depressant in coal flotation (Ozbayoglu, 1987). For pH control lime-soda ash, caustic soda and mineral acids are generally used.

This paper reports the results on the study to optimise the operating process variables of the flotation, which may be implemented in large scale flotation cell to utilise the vast resources of low rank coal in India in a better way.

2. EXPERIMENTAL

2.1. Material

About 2 tonne of run-of mine non-coking coal sample from Jagannath mine, Talcher coal field, India was collected and used for this investigation. The as-received sample was crushed to -10mm using laboratory Blake jaw crusher and then it was again crushed to –1mm size using laboratory roll crusher. The roll crusher product was thoroughly mixed and representative sample of approximately 5kg each was prepared. Then each representative sample was ground by laboratory ball mill (9"x9"size) to below 100μm in closed circuit wet grinding method at 300% load as per the standard method. The standard BSS sieve was used for size distribution of ground product. 100+75, -75+45, -45μm fractions were generated and proximate analysis of each fraction was carried out. The results of these samples are given in Table 1.

Table 1
Size and proximate analysis of ground coal sample

Size, μm	Weight, %	Moisture, %	V. M., %	Ash, %	F. C., %
-100 + 75	21.2	3.99	27.45	39.27	33.28
-75 + 45	20.0	4.31	26.69	37.18	36.13
-45	58.8	3.70	23.35	41.54	35.11
Feed	100.0	3.80	24.88	40.18	34.40

The chemical analysis of ash from coal was carried out and is given in Table 2. The mineralogical analysis of silica particle in coal was done by image analyser to know the liberation size of ground coal and data is given in Table 3. Commercial grade light diesel oil was used as collector whereas MIBC supplied by National Organic Chemical Ltd. Bombay, India as frother. Commercial grade sodium silicate was used as silica depressant. Analytical grade methanol, ethanol, butanol and octanol were used as reagents for pretreatment of coal.

Table 2
Chemical analysis of ash from rom coal

Constituents	Percentage
SiO_2	54.49
Al_2O_3	24.14
Fe_2O_3	12.21
TiO_2	1.79
CaO	2.23
MgO	1.21
Others	3.92

Table 3
Mineralogical analysis of silica particle in coal

Size, μm	Percentage
0 - 10	45.77
10 - 20	34.43
20 - 30	14.84
30 - 40	2.68
40 - 50	1.65
50 - 60	0.61

2.2. Pretreatment of oxidised coal

Oxidised non-coking coal was pretreated with, four aliphatic alcohols i.e., methanol, ethanol, butanol and octanol separately. These studies were carried out using Denver D-12 sub-aeration flotation cell with 1 litre capacity of cell. About 80g of coal sample was taken in each experiment and made 40% of solid concentration with water. Then it was treated with sodium silicate for 20 minutes at 1000rpm for silica dispersion and depression. Then required dosages of alcohol was added and agitated in the cell for 20 minutes. For each alcohol, separate studies were carried out. These pretreated coals slurries were subjected to flotation study. In each case the required amount of dosage of collector was added and conditioned for 10 minutes. The additional water was added to bring down the solid concentration up to 10% and then flotation was carried out at natural pH(6.0) after adding the required dosage of frother. One blank flotation test was carried out without using any alcohol for pretreatment of coal surface. The concentrate and tailings were collected separately. These samples were dried and weighed and analysed for ash according to ASTM method.

2.3. FTIR study

The samples used for FTIR analysis were ground and pellets were prepared using spectroscopic grade KBr by standard procedure. Different samples, viz, r.o.m. coal, r.o.m coal pretreated with ethanol and also flotation product obtained with pretreated coal were subjected to FTIR studies in order to know the oxidative property. Model FTIR-5300, supplied by Japan Spectroscopic Co. Ltd. 2967-5, Ishikawa-CHO, Hachioji city, Tokyo, Japan was used for this study.

2.4. Electro-kinetics study

Electro-kinetics study of macerals i.e., vitrain, durain and fusian and bulk sample were carried out for further verification regarding the oxidation of coal surface. Electro-kinetics studies have been conducted by measuring the electrophoretic mobilities of fine coal particles in water suspension by Electrophoretometer, Rank Brothers Ltd. Cambridge, England using KCl as electrolyte.

2.5. Flotation tests

As the response of ethyl alcohol towards de-oxidation of coal surface was better in comparison with other aliphatic alcohols, it was used in all flotation experiments as pretreatment reagent for de-oxidation. All flotation tests were carried out using laboratory Denver D-12 sub-aeration flotation machine. The volume capacity of the cell is 10L. About 800g of sample was taken in each experiment. The sample was made 40% solid concentration with water and treated with sodium silicate for both depression and dispersion of silica particles. Then it was pretreated with required amount of ethyl alcohol. The required amount of collector was added and conditioned. The solids concentration was brought down to 10% by adding additional water in the cell. Then the required amount of frother was added and conditioned for two minutes. Then the flotation was carried out using the induced air. The experiments were carried out at different operating parameters. The flotation concentrate and tailings were collected separately in each experiment and both were dried, weighed and analysed for ash according to ASTM standard.

3. RESULTS AND DISCUSSION

The results of the flotation study using different aliphatic alcohol as pretreatment reagent are given in Table 4. It has been observed that aliphatic alcohols are excellent active agent to enhance the floatability of oxidised non-coking coal. The floatability of oxidised non-coking coal could be enhanced due to dissolution of oxidised layer by aliphatic alcohol. It has also been observed that ethanol gives better pretreatment effect than other alcohols. By pretreatment with ethanol, the flotation test gives 20.0% ash and 90.5% combustible recovery. Based on this the optimisation study, the dosage of ethanol was carried out. The result of this study is reported in in Figure 1. It has been observed that the requirement of one kg of ethanol per tonne of r.o.m. coal is sufficient with respect to combustible recovery and ash content of the flotation concentrate.

Table 4
Comparison of flotation results using different alcohols as pretreated reagents

Sl. No.	Alcohol	Details	Weight recovery, %	Ash, %	Feed ash, %	Combustible recovery, %
1	Nil	Conc.	57.2	21.0	36.28	70.91
		Tailings	42.8	56.7		
2	Methanol	Conc.	75.0	23.9	35.72	88.79
		Tailings	25.0	71.2		
3	Ethanol	Conc.	72.4	20.0	36.00	90.51
		Tailings	27.6	78.0		
4	Butanol	Conc.	64.0	20.2	35.97	79.78
		Tailings	36.0	64.0		
5	Octanol	Conc.	72.0	21.9	35.83	87.63
		Tailings	28.0	71.65		

MIBC: 0.5ml/kg, Diesel oil: 1.0ml/kg, Sodium silicate: 1.0g/kg
Alcohol: 1.0ml/kg, Solid concentration in the slurry: 10.0%, Rpm: 1000

FTIR spectroscopy has evoked a lot of interest now-a-days for its ability to throw information regarding the surface conditions and the characteristics of coal (Ye *et al.*, 1988; Zimmerman, 1964; Frumkin 1926). Enhancement of floatability in the experiment was explained through FTIR study. The results of this study are shown in Figure 2. It is established that the hydrophilicity index may be expressed in terms of surface functional groups as follows(Ye *et al.* 1988; Yuh *et al.*, 1983; Painter, 1983);

$$\text{Hydrophilicity Index} = \frac{\text{ล}(\text{-COOH}) + \text{ล}2(\text{-OH})}{\text{ล}(\text{R-H}) + \text{ล}(\text{Ar-H})} \qquad (1)$$

Where ล(-COOH), ล(-OH), ล(R-H) and ล(Ar-H) are the values of the absorption intensity. Using such a definition the value of hydrophilicity from FTIR spectra has been computed to be as 1.51, 0.92 and 0.12 in case of r.o.m., ethanol treated and floated coal respectively.

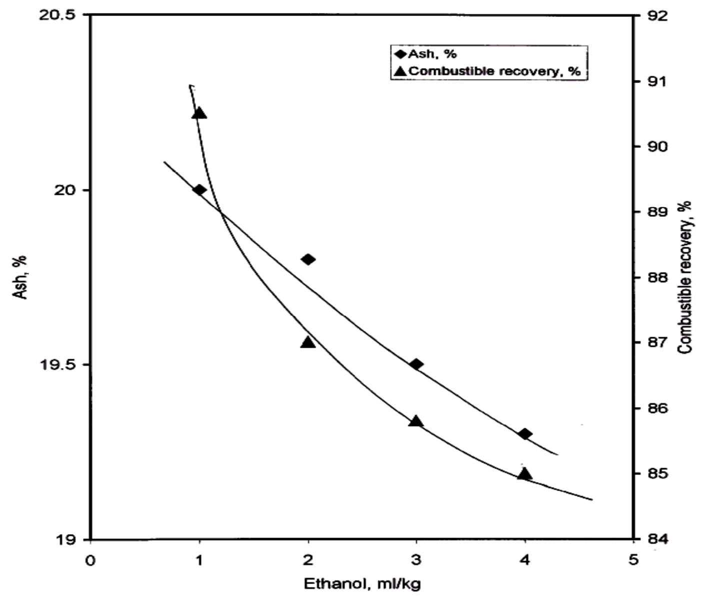

Figure 1. Effect of ethanol

The aromatic hydrogen (Ar-H) and aliphatic hydrogen (R-H) stretching frequency mode, generally observed around the region of 3020 to 1450cm^{-1}, attributes to the hydrophobic characters of the coal. It has been observed from Figure 2 that the intensity of absorption (Ar-H, R-H) on r.o.m. coal is less compared to ethanol treated and floated coal. The hydroxyl and carboxyl groups describing the oxidative nature of coal are generally observed much more in case of r.o.m. coal showing predominance of these oxygen bearing groups. The peak intensity is seen to be low in case of 2 and 3 showing certain extent of hydrophobicity attainment which has been subsequently proved in flotation with respect to grade and recovery.

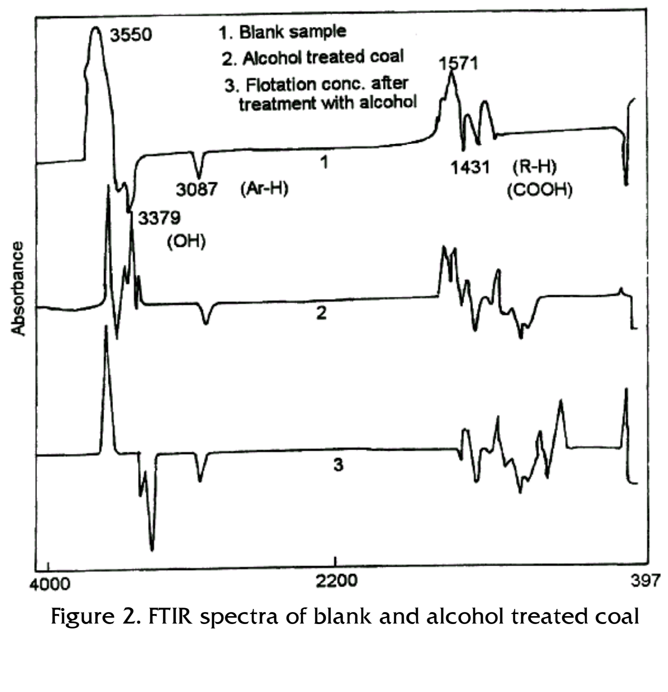

Figure 2. FTIR spectra of blank and alcohol treated coal

Since the chemical composition of coal macerals differs, the various coal macerals respond differently to flotation when coal is subjected to oxidation. In general, the vitrain of un-oxidised coal should have point of zero charge (PZC) at higher pH than that of other macerals due to less mineral content in the coal matrix. Bennett *et al.* (1983) has also reported that the grains which consisted entirely of vitrinite, clarite, vitrinertinite or trimacerite have similar flotabilities provided that they have no visible mineral matter. The results of zeta potential study is shown in Figure 3. It has been observed that from experimental data that vitrain shows PZC at lower pH compared to the other macerals, indicating that the surface of the coal was oxidised and all such coal has been used for the subsequent experiments. The PZC of bulk sample is found at pH 3.8. It has been reported that when oxidation time increases, the iso-electric point decreases and the negative zeta potential increases. The point of zero charge of non-oxidised coal is more than that of oxidised coal (Sarikaya *et al.*, 1995). In general, the rate of oxidation of low rank coal is faster than high rank coal. With respect to petrographic component, oxidation increases in the order of vitrain, clarain, durain and fusain in coals of all ranks (Ozbayoglu, 1987).

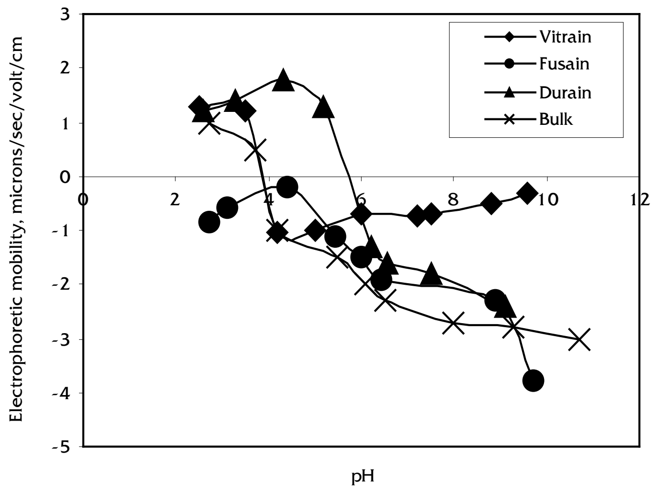

Figure 3. Electrokinetics of coal macerals and bulk sample

It is observed from Table-1 showing the size distribution of ball mill product that the ash percentage in each fraction is almost similar to each other, hence it is difficult to beneficiate by simple classification. The chemical analysis of ash from coal was carried out and given in Table 2 and it indicates that the silicate and alumina composite clay minerals are more in the composition of gangue minerals. It helps to estimate the dosage of sodium silicate for dispersion and depression. The mineralogical analysis of silica particles in coal was done by image analyser to know the liberation size of ground coal given in Table 3. Most of silica particles are free from coal particles in below 30μm.

The effect of collectors on coal flotation has been reported by many authors for various types of coal with different petrographic structures (Klassen, 1963; Viasova *et al.*, 1962; Horsley *et al.*, 1951; Onlin *et al.*, 1989; Aplan, 1993). The effect of diesel oil dosage on flotation performance is shown in Figure 4. It has been observed that the results on the effect of collector in the present investigation are in agreement with the reported by others. The selectivity is better at lower collector dosage in comparison with higher dosages. From this data, both combustible recovery and grade of product are more acceptable at the collector level of 1.0ml/kg of coal, though combustible recovery at 2.5ml/kg level is maximum, but the ash content is high. Depending on the rank the amount of collector consumption varies from 0.2-2.5kg/tonne ranging from the high bituminous coal to oxidised non-coking coal (Aplan, 1976). In this case the amount of collector dosage is high due to average size of particle is less in comparison with normal conventional coking coal flotation and also due to the higher percentage of ash.

Flotation results obtained using MIBC as frother is shown in Figure 5. The effect of frother dosage shows that combustible recovery increases with increasing of frother concentration. The frother has lot of significant effect on mean bubble size which plays an important role in determining the flotation performance and the bubble size be manipulated by varying frother dosage. But to maintain the acceptable quality of product, the frother dosage of 1ml/kg has been decided to help constant for the experiments which varying other parameters. Higher concentration of frother forms foam type froth, which prevents the drainage of gangue particles through the froth bed and increases the ash content of concentrate. Foam type froth comes under tight and close knit froth, where small bubbles remain very close to each other but do not coalesce easily to form big bubbles and collapse to the atmosphere after a pretty long time, which causes more entrainment of gangue minerals in the froth (Bhaumik, 1989).

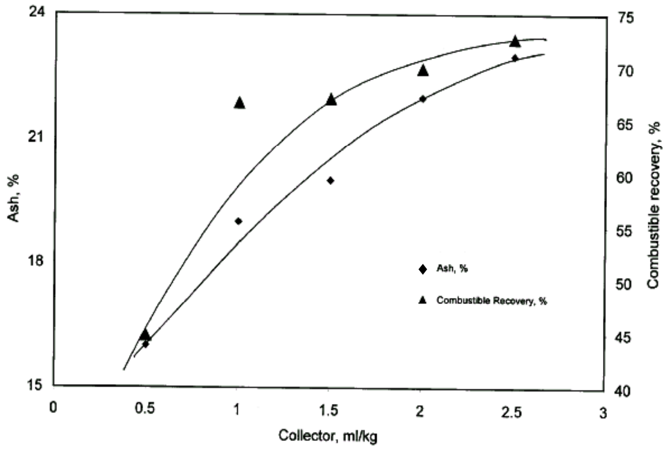

Figure 4. Effect of collector

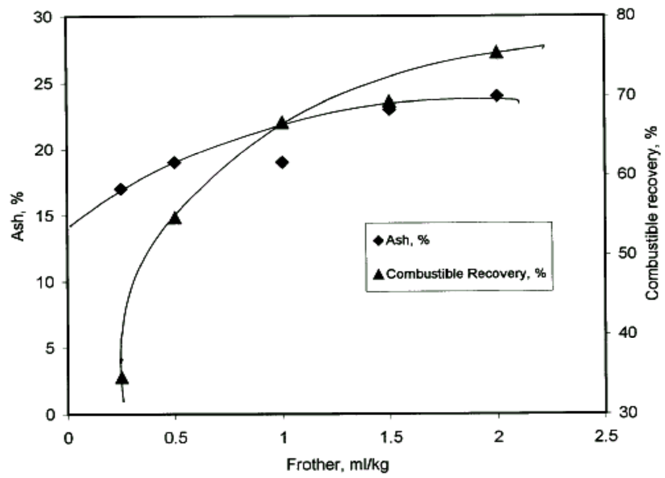

Figure 5. Effect of frother

As the high ash coal has been ground to below 100µm size, the generation of slime is obvious. The slime particles of alumina-silicate clay are mostly dominant in this gangue minerals which strongly affects the flotation performance due to slime coating of clay particles on the coal surface (Jowett *et al.*, 1956). The slime clay particles also affect the flotation of coarse particles as well as increase the ash content in the flotation concentrate. However, coal hydrophobicity can be restored by simply adding a dispersant. The effect of sodium silicate as depressant is shown in Figure 6. This figure shows that both combustible recovery and grade are better acceptable at the dosage of 1.0g/kg of depressant whereas the good quality of product can be obtained at higher rate of depressant. By addition of depressant, negative values of electrokinetic potential increases, which is enough to prevent clay particles from coagulating on to the coal surface. At the higher dosage of depressant which causes the depression of locked particles of silica mineral and coal, hence the grade of concentrate increases and simultaneously the recovery decreases. Therefore, the effect of clays on flotation can be eliminated by using dispersing agents.

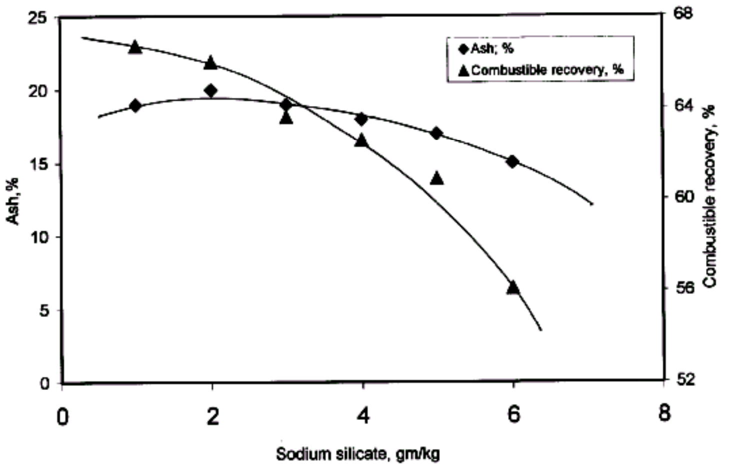

Figure 6. Effect of depressant

The effect of impeller speed on flotation performance is shown in Figure 7. From these results, there is a maximum recovery with quality of product at 1000 revolution per minute of impeller speed. The entrained air rate increases with revolution per minute leading to generation of increased froth-volume and hence, increased recovery. Too low agitation gives inefficient mixing and dispersion of oily collector and poor performance on collision between particle and bubble (Aplan, 1976). Excessively high speed can decrease the effective air dispersion, which may decrease the recovery of concentrate (Aplan, 1976).

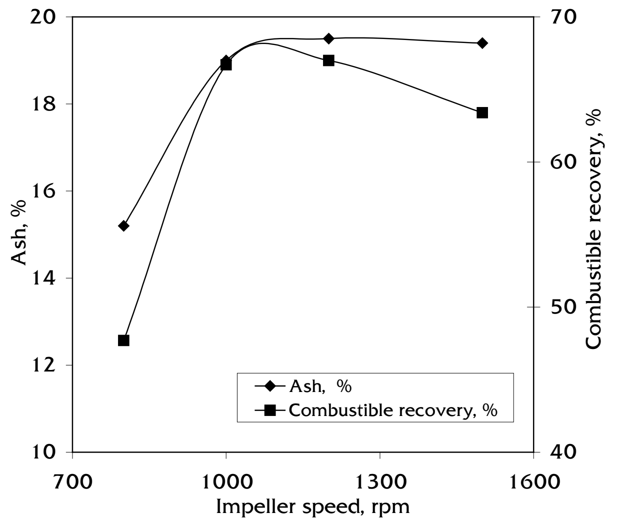

Figure 7. Effect of impeller speed

Flotation results in different conditioning time is shown in Figure 8. For the benefits in both combustible recovery and quality, 10 minutes. conditioning time gives better results than others. Initially with an increased conditioning time, the dispersion of collector (diesel oil) droplets in the coal water slurry increases and this helps in a better attachment of the collector film on the surface of the particles by adsorption making it more hydrophobic (Aplan, 1976). As the conditioning time is further increased some of the fine mineral material may also adsorb the oily collector on their surface, which will make them hydrophobic (Aplan, 1976).

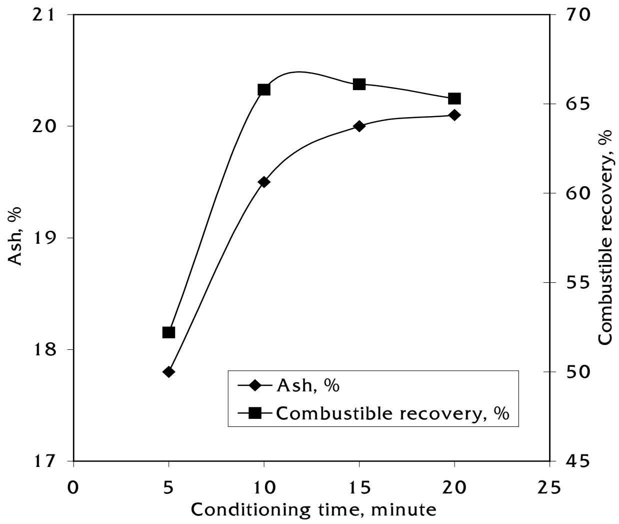

Figure 8. Effect of conditioning time

The effect of the pH was studied and is shown in Figure 9. At pH 6.0, the recovery and ash percentage is better. It has been reported that the floatability of coal is maximum when pH is between 6.0 and 8.0 (Zimmerman, 1948; Bordon, 1962). It has also been observed in this work that the point of zero charge (PZC) of oxidised coal is at pH 4.4. After pretreatment of coal surface with ethanol before flotation, iso-electric point might have shifted to the alkaline side at pH 6.0 of the slurry, hence the grade of concentrate is better than that obtainable at other pH.

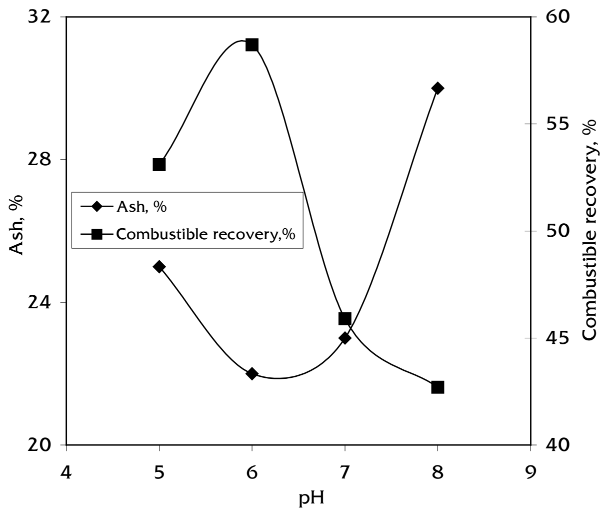

Figure 9. Effect of pH

4. CONCLUSIONS

The role of oxidation on hydrophilicity index can be visualized through the measurement of hydrophilicity index using FTIR data and electro-kinetics study. The floatability of coal could be enhanced using aliphatic alcohols. Ethanol pretreated flotation result can give in better performance with respect to grade and recovery, due to dissolution of the oxidized layer by alcohol. The consumption of chemical reagents are high in comparison with coking coal due to low hydrophobicity of oxidised coal surface and fine size of particles.

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