

Eco Friendly and Cost-Effective Reagent for Coal Flotation

N.Vasumathi^{1)*}, T.V.Vijaya Kumar¹⁾, S.Subba Rao¹⁾, S.Prabhakar¹⁾, G.Bhaskar Raju¹⁾,
S.Shiva Kumar²⁾ and Uma Raman²⁾

1) CSIR-National Metallurgical Laboratory Madras Centre, CSIR Madras Complex, Taramani, Chennai
600113, India.

2) Somu Organo Chem Pvt. Ltd., Bangalore 560076, India.

* Corresponding Author Email Id: vasumatisamy@gmail.com;

Abstract

Conventionally, diesel in combination with a frother is used widely in flotation of coal fines. With the continuous price escalation of petroleum products and their negative impact on environment, attempts were made to formulate an eco-friendly single reagent to replace diesel-frother system without affecting the flotation performance. Laboratory flotation tests were carried out, on a coking coal sample from eastern India, that analyzed 25.67% ash and 53.97% fixed carbon using a series of reagents developed. Among them, the performance of Sokem 590C derived from a vegetable oil was found to be encouraging. Concentrate assaying 11.77% ash and 66.40% fixed carbon was obtained with yield of 56.57%. Moreover, the reagent is biodegradable and eco friendly. Based on favorable kinetics and encouraging test results, plant trials were conducted at a coal preparation plant and the superiority of this reagent was demonstrated.

Keywords: Coal flotation; Diesel; Frother; Single reagent, Biodegradable collector.

Introduction

Coal is a heterogeneous degradation product of vegetable and mineral matter, fossilized through oxidation [1]. The appearance and properties of coal mainly depend on the nature of its origin and the physical and chemical changes occurred after deposition. It is susceptible to weathering and as a result oxygenated functional groups will be formed on the surface, which in turn alter the degree of natural hydrophobicity. The natural hydrophobic character is an important criterion to arrive at the coal process flowsheet. The removal of shale bands present within the coal seams [2] is a major challenge in coal cleaning process when compared to other forms of fuels [3]. Modern mechanized coal mining techniques generate large amount of fine particles that have to be recovered in coal washing plants [4]. Fine coal processing has always been a problematic and costlier than the cleaning of coarse coal [5]. Froth flotation technique is an effective and best available technique for

cleaning fine coal with particle size less than approximately 0.5mm [6-8]. Flotation is a solid-solid separation process in aqueous solution based on the difference of hydrophobic character of the substances to be separated [9]. In flotation, fine particles show lower flotation rate, resulting in low flotation recovery [10]. Flotation utilizes the differences in the surface properties mainly the hydrophobic character [11]. However, the hydrophobicity or contact angle of coal is varied according to the heterogeneity of surface [12-14]. The separation efficiency depends on the wettability difference between the coal-rich and mineral-rich particles of the coal slurry in the flotation circuit. In froth flotation, coal particles are subjected to a suitable hydrocarbon (liquid) treatment [15] to alter the hydrophobicity, enhance recovery and /or improve selectivity [16]. Conventionally, the collector used in most of the Indian coal washeries is diesel oil in combination with different frothers. The dosage of collector and frother has significant effect on flotation performance [17]. Since the diesel oil is non-polar oil, its disposal in an environmentally acceptable manner is challenging task. Hence, a replacement for diesel was initiated. Vegetable oils namely, crude soybean and olive oils have been reported as collectors for the recovery of coal fines [18]. Colza oil was used as collector for the recovery of high-calorific and low ash content coal [19]. Polanga and Mahua oils were used as collectors to enhance floatability of high ash Indian non-coking coal [20]. A replacement for this diesel-frother system has been attempted by utilizing vegetable oils & biodiesel [19]. But, all these studies were confined to laboratory scale only. In this work, vegetable oil based and eco-friendly reagent was developed as a replacement for diesel-frother system. This single reagent developed was tested for its efficiency at laboratory scale. Further, flotation kinetics were also carried out for both single & dual reagent system which help to explain the behavior of floated particles and to measure the influence of different process parameters of flotation [21]. After encouraging results are obtained at laboratory scale, trials were undertaken at industrial scale in a coal preparation plant with a flotation circuit capacity to process 30t/h.

Experimental work

Material preparation

The coking coal fines from Jharia region in eastern India were obtained for flotation tests. The proximate analysis, particle size and ash content of the coal sample are presented in Tables 1 and 2 respectively.

Table 1. Proximate Analysis of coal sample

Sample	Ash (%)	Moisture (%)	Volatile Matter (%)	Fixed Carbon (F.C) (%)
Coal (feed)	25.67	0.85	19.51	53.97

Table 2. Particle Size & Ash Analysis of coal sample

Size (µm)	Wt. retained (%)	Ash (%)
+500	16.61	32.16
-500+355	13.58	26.95
-355+212	18.46	25.67
-212+100	22.11	23.71
-100	29.24	25.27

The coal sample assayed 25.67 % ash and its d_{80} is 421 µm. The +500 µm size fraction has the highest ash content of 32.16 % as compared to the other size fractions.

Petrology

The petrology studies of the coal sample were carried out using petrological ore microscope and the details are given in Table 3.

Table 3. Petrological Analysis of coal

Sample	Vetrinite (%)	Exinite (%)	Inertinite (%)	Mineral matter (%)
Coal (feed)	45.8	0.8	26.0	27.4

X-ray diffraction studies

The X-ray diffractogram (Seifert XRD 3003 TT, GE Inspection Technologies, Germany) of the coal sample

indicates the presence of quartz and kaolinite gangue. The characteristic copper-K α X-ray radiation with 1.54 Å wavelength was used in this diffraction study. The XRD of the float product i.e. coal concentrate obtained using Sokem 590C, presented in Fig. 1, clearly shows that the intensity of the quartz and kaolinite peaks is comparatively weaker than that of the feed. Thus it is evident that the ash content in the float is considerably reduced.

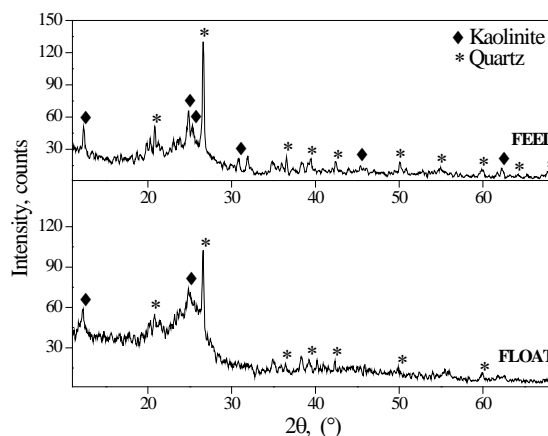


Fig. 1. X-ray diffractogram of coal feed and float

Flotation Methodology

The laboratory scale experiments are conducted using Denver flotation machine, D-12 with a cell volume of 3.0 L. The coal slurry (12% solids by weight) is conditioned with diesel and frother / single reagent for 3 minutes each. All the experiments are conducted at natural pH and the solids are kept in suspension by the impeller rotating at a speed of 1200 rpm. The collected float and tailings are dewatered, dried, weighed and subjected to ash analysis. The combustibles recovery (CR), ash rejection (AR) and the efficiency index (EI) are calculated from the weight recovery / yield and ash values for each test using the following equations respectively.

$$\%CR = \frac{M_c(100 - A_c)}{M_f(100 - A_f)} \times 100 \quad (1)$$

$$\%AR = 1 - \left(\frac{M_c A_c}{M_f A_f} \right) \times 100 \quad (2)$$

$$EI = (CR + AR) - 100 \quad (3)$$

where, A_c : ash content in concentrate (%)

A_f : ash content in feed (%)

M_c : Mass of concentrate (g)

M_f : Mass of feed (g)

The diesel and a proprietary frother were obtained from a chemical supplier. A series of single reagents (Sokem 580C series & Sokem 590C) used for evaluation studies are proprietary chemicals provided by M/s Somu Organo Chem. Pvt. Ltd., (SOCPL) Bangalore, India. Their response was evaluated based on the yield and ash content of the float. The results are compared with those obtained for diesel-frother reagent system.

Plant trials were conducted in a flotation plant for validating the findings obtained at laboratory scale studies.

Results and discussion

Flotation tests using a series of single reagents

A series of single reagents (Sokem 580C series & Sokem 590C) was tried for coal flotation at different dosages of each reagent. The reagent dosage corresponding to the maximum yield of the float with ash ranging from 11% to 16% are considered and the results are given in Table 4.

Table 4. Results using various single reagents of Sokem 580C series & Sokem 590C

Single reagent	Dosage (kg/t)	Yield (%)	Ash (%)	C.R (%)	E.I
Sokem 583C	0.3356	62.94	14.95	71.64	34.40
Sokem 584C	0.3524	64.47	16.00	73.11	33.33
Sokem 586C	0.1742	64.14	15.89	72.41	32.44
Sokem 587C	0.2693	62.50	15.76	71.98	35.31
Sokem 589C	0.0762	54.82	14.38	63.10	32.32
Sokem 590C	0.0775	56.57	11.77	66.02	38.73

From the results, it is apparent that Sokem 590C yields 56.57% float with the lowest ash of 11.77%. It appears to be more economical in terms of reagent consumption with better separation efficiency. Hence, more elaborate flotation tests were carried out using Sokem 590C and the results are given in Table 5.

Table 5. Flotation tests results using single reagent Sokem 590C

Diesel (kg/t)	Frother (kg/t)	Yield (%)	Ash (%)	CR (%)	EI
0.363	0.033	47.45	13.31	55.47	31.04
0.454	0.049	52.36	14.00	60.08	30.50
0.545	0.065	67.86	16.60	76.40	32.96
0.726	0.065	81.37	18.84	87.18	23.96
0.726	0.098	84.92	18.94	91.43	26.34

As the dosage of Sokem 590C is increased, the yield of float increased from 51.27% to 77.16% with concomitant increase in ash content in the float and the combustibles recovery. However, separation efficiency was the highest at the dosage of 0.0775 kg/t. At higher dosages, relatively lower separation efficiency indices could be attributed to reporting of poorly liberated macerals into the float.

Flotation test using diesel and frother

The flotation tests were carried out using diesel and a commercial frother and the results are given in Table 6, which can be used to compare and analyze the performance and efficiency of the single reagent.

With diesel-frother system, the float assayed 13.31% ash at 47.45% yield. Further increase in dosages of diesel-frother resulted in higher yield and also higher ash content in the float, which could again be attributed to the poorly liberated macerals reporting to float. But efficiency indices in case of Sokem 590C are relatively higher than those of diesel-frother system. This implies that better separation and selectivity could be achieved using Sokem 590C vis-a-vis diesel-frother system.

Table 6. Flotation test results using diesel and frother

Diesel (kg/t)	Frother (kg/t)	Yield (%)	Ash (%)	CR (%)	EI
0.363	0.033	47.45	13.31	55.47	31.04
0.454	0.049	52.36	14.00	60.08	30.50
0.545	0.065	67.86	16.60	76.40	32.96
0.726	0.065	81.37	18.84	87.18	23.96
0.726	0.098	84.92	18.94	91.43	26.34

The yield versus ash curve for both the reagent systems is plotted and is shown in Fig. 2.

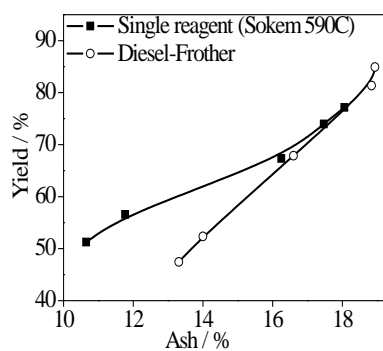


Fig. 2. Yield Vs Ash plot for two different reagent systems

The proximate analysis of a final float product obtained using single reagent (0.0775 kg/t) & diesel-frother system (0.363 kg/t and 0.033 kg/t respectively) at near-equivalent ash values were subjected to complete proximate analysis and the results are shown in Table 7.

Table 7. Proximate Analysis of float using single reagent & diesel-frother system

Sample	Ash (%)	Moisture (%)	Volatile Matter (%)	F.C (%)
Float (Sokem 590C)	11.77	0.89	20.94	66.40
Float (Diesel-frother)	13.31	0.97	20.97	64.75

Flotation kinetics study

Flotation kinetics studies were carried out on coal using both the reagent systems. The float samples were collected at various time intervals and analyzed for its ash content. The results of the same are used in the calculation of the rate constant as presented in Fig. 3. The flotation rate equation is expressed as shown below [9]:

$$\frac{dC}{dt} = -kC^n \quad (4)$$

where, C is the concentration of solids, t is the flotation time, n is the order of the process and k is the rate constant.

If n=1, the above equation (4) becomes

$$\frac{dC}{C} = -kdt \quad (5)$$

On integration, equation (5) gives rise to

$$\ln \left(\frac{C_0}{C} \right) = kt \quad (6)$$

where, 'C₀' is the initial concentration of coal in the flotation cell, 'C' is the concentration of coal remaining in the cell at the given time 't' and 'k' is the flotation rate constant.

If the values for 'ln(C₀/C)' are plotted against flotation time 't', the slope of the straight line obtained is the flotation rate constant 'k'. The data obtained for the calculation of 'k' is shown in Fig. 3 for coal on using both single reagent (Sokem 590C) and the conventional diesel & frother system.

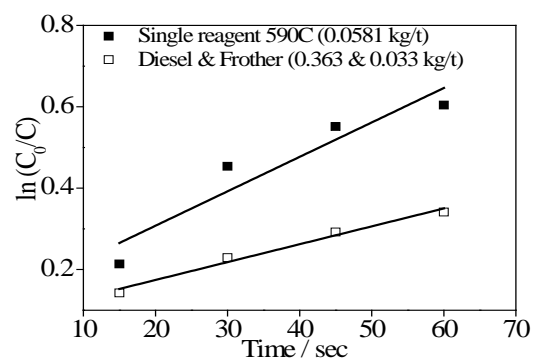


Fig. 3. Flotation rate constant plot for two different reagent systems

The rate of flotation of solid particles determines the percentage recovery of the particles which can be obtained during flotation. From Fig. 3, it is clear that Sokem 590C provides faster flotation kinetics of coal ($k=0.00846 \text{ s}^{-1}$) as compared to that of the diesel & frother regime ($k=0.00436 \text{ s}^{-1}$).

Plant trials using single reagent

The results of conventional plant practice with diesel and frother system are given in Table 8. Trials were conducted with Sokem 590C in the same plant flotation circuit of 30 t/h capacity. The results of the same are given in Table 9.

Table 8. Results of plant trials using diesel & frother

Trial period	Shift	Diesel/frother dosage (ml/min)	Feed Ash (%)	Float Ash (%)	Tail Ash (%)	Yield (%) (fine coal rec.)
Day 1	A	450/50	24.97	14.11	40.03	
	B	500/50	28.43	18.73	38.88	
	C	500/50	27.97	18.81	37.18	53.37
Day 2	A	500/50	28.71	18.22	35.62	
	B	500/50	28.17	19.15	35.69	
	C	500/50	28.30	21.25	42.90	50.87

Table 9. Results of plant trials using Sokem 590C

Trial Period	Shift	Sokem 590C dosage (ml/min)	Feed Ash (%)	Float Ash (%)	Tail Ash (%)	Yield (%) (fine coal rec.)
Day 1	A	120	25.94	13.94	38.37	
		120	26.14	13.98	48.18	
		120	33.38	18.56	46.34	53.99
	B	120	22.93	13.73	37.44	
		120	23.10	13.63	37.20	
		120	23.87	13.81	40.02	60.88
		120	25.91	14.86	38.98	
	C	120	23.34	13.98	33.01	
		120	20.24	13.82	39.36	58.66
		120	24.51	14.18	37.03	

From the plant trials results, it indicates that single reagent Sokem 590C is very effective in terms of yield and ash content in the float. It is observed that the reagent is more effective in collecting fine and ultrafine coal particles, rather than the coarser particles.

The consumption of Sokem 590C is much less compared to that of diesel being used in the plant. This single reagent doesn't have adverse effect on the downstream operations in the plant such as thickener water quality. Above all, the single reagent addition at a single point also makes the operation easier.

Cost benefit analysis

The cost and benefits analysis was carried out to compare the economic viability in using single reagent in place of diesel-frother system. The reagents cost estimation comparison for treating one tonne of coal is given in Table 10.

Table 10. Cost benefit analysis for single reagent, Sokem 590C and diesel-frother system

Parameters	Values	Cost (INR)
Feed rate (tonne/hour)	1.0	--
Diesel consumption (kg/t)	0.80	50.00
Frother consumption (kg/t)	0.092	12.00
Sokem590C consumption (kg/t)	0.226	33.80

It is evident that the cost of single reagent is cheaper by about 47% per tonne of feed as compared to diesel-frother system. The throughput capacity of the flotation circuit in the plant where trials were carried out is 30 t/h. So, per day, the reagent consumption cost for Sokem 590C would be 24336.00 INR while that of diesel-frother system would be 51840.00 INR. This is over and above the reduced capital expenditure and operational simplicity.

Conclusion

The new single reagent Sokem 590C was evaluated for its efficiency on coal flotation both in laboratory bench scale studies and in an operating coal preparation plant. It was found that single reagent was superior to the conventional diesel-frother system, in terms of yield and ash content. Flotation results also indicate that the single reagent Sokem 590C is effective in improving the flotation performance of fines in the flotation circuit. Adopting single reagent Sokem

590C proves to be more efficient in coal flotation as borne out from the flotation kinetics studies as well. Considering the plant operation point of view, handling single reagent system is much easier than compared to two-reagents system. This single reagent Sokem 590C proves to be cost effective as compared to diesel-frother system. Moreover, it is non-petroleum and vegetable oil based, biodegradable and environmental friendly.

Acknowledgements

The authors wish to thank The Director, CSIR-NML for his support and permission to publish this work and M/s Somu Organo Chem Pvt. Ltd., for their logistical support.

References

- i. J. A. Gutierrez-Rodriguez, R. J. Jr. Purcell and F. F. Aplan, Estimating the hydrophobicity of coal, *Colloids and Surfaces*, Vol. 12, pp. 1-25 (1984).
- ii. Zhenghe Xu, Jainjun Liu, J. W. Choung and Zhiang Zhou, Electrokinetic study of clay interactions with coal in flotation, *Int. J. of Mineral Processing*, Vol. 68, pp. 183-186, (2003).
- iii. B. Ambedkar, R. Nagarajan and S. Jayanti, Investigation of High-frequency, high-intensity ultrasonics for size reduction and washing of coal in aqueous medium, *Industrial & Engineering Chemistry Research*, Vol. 50, No.23, p.13210-13219 (2011).
- iv. Shaoxian Song, Experimental studies on hydrophobic flocculation of coal fines in aqueous solutions and flotation of flocculated coal, *Int. J. Oil, Gas and Coal Technology*, Vol. 1, pp.181-193 (2008).
- v. C. Andrew Mohns, Effect of Particle Size on Coal Flotation Kinetics, [Dissertation], Queen's University, Kingston, Canada, 1997.
- vi. R. H. Yoon, G. H. Luttrell and R. Asmatulu, Extending the upper particle size limit for coal flotation, *The Journal of The South African Institute of Mining and Metallurgy*, pp.411-415 (2002).
- vii. C. N. Bensley and S. K. Nicol, The effect of mechanical variables on the flotation of coarse coal, *Coal preparation*, Vol 1, pp.189-205 (1985).
- viii. S. Koca, Y. Bektas and H. Koca, Contact angle measurement on lignite surface, *Proceedings of the XXV International Mineral Processing Congress (IMPC)*, Brisbane, Australia, 2010, pp. 2049-2053.
- ix. J. S. Laskowski, *Coal Flotation and Fine Coal Utilization*, Elsevier, Amsterdam, 2001.
- x. Daniel Chipfunhu, Massimiliano Zanin and Stephen Grano, The dependency of critical contact angle for flotation on particle size-Modeling the limits of fine particle flotation, *Minerals Engineering*, 24(2011), pp.50-57.
- xi. Li Ping Ding, Investigation of Bituminous coal hydrophobicity and its influence on flotation, *Energy & Fuels*, Vol. 23, pp.5536-5543 (2009).
- xii. B. J. Arnold and F.F. Aplan, The hydrophobicity of coal macerals, *Fuel*, Vol. 68, No.5, pp. 651-658(1989).
- xiii. G.A. Brady and A.W. Gauger, Properties of coal surfaces, *Journal of Industrial Engineering and Chemistry*, Vol. 32, pp.1599-1604 (1940).
- xiv. A. Gosiewska, J. Drelich, J.S. Laskowski and M. Pawlik, Mineral matter distribution on coal surface and its effect on coal wettability, *Journal of Colloid Interface Science*, Vol. 247, No.1, pp.107-116 (2002).
- xv. F. Boylu and J.S. Laskowski, Rate of water transfer to flotation froth in the flotation of low-rank coal that also requires the use of oily collector, *Int. J. of Mineral Processing*, Vol. 83(2007), pp.125-131.
- xvi. F.F. Peng, Surface energy and induction time of fine coals treated with various levels of dispersed collector and their correlation to flotation responses, *Energy & Fuels*, Vol. 10(1996), pp.1202-1207.
- xvii. S. Dey and S. Pani, Effective processing of low-volatile medium coking coal fines of Indian origin using different process variables of flotation, *International Journal of Coal Preparation and Utilization*, Vol. 32 (2012), pp. 253-264
- xviii. M.I. Alonso, C. Castano and A.B. Garcia, Performance of vegetable oils as flotation collectors for the recovery of coal from coal fines wastes, *Coal Preparation*, Vol. 21, No.4, pp.411-420 (2000).
- xix. W. Xia, J. Yang and C. Liang, Improving oxidized coal flotation using biodiesel as collector, *Int. J. of Coal preparation and Utilization*, Vol. 33, pp. 181-187 (2013).
- xx. B. Das and P.S.R. Reddy, The utilization of non-coking coal by flotation using non-conventional reagents, *Energy Sources, Part A*, Vol. 32, pp.1784-1793 (2010).
- xxi. A.P. Chaves and A.S. Ruiz, Considerations on the kinetics of froth flotation of ultra fine coal contained in tailings, *Int. J. of Coal Preparation and Utilization*, Vol. 29, pp. 289-297 (2009).