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RESEARCH PAPER

Investigating the Effect of Zero Prewetting Time on Rougher Flotation of Coal Tailings

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Abstract: *The beneficiation of coal tailings is usually difficult by common oily collectors in the flotation process, so it is necessary to use a suitable method for clean coal recovery from coal tailing dams. Thus, this study was aimed to investigate the behavior of dissolved air flotation by zero prewetting time for the clean coal recovery and to optimize the conditions of zero prewetting time for an effective flotation. In this regards, the effects of the process parameters, i.e., pH, frother type, collector type on the rougher flotation recovery of coal tailings were assessed and optimized. Additionally, Fourier transform infrared (FTIR) spectroscopy was used to understand the functional groups of oily collectors on the surface of floated products. The findings indicated that the frother type and the interactive effects between the type of frother and collector had the most effect on the performance of flotation. It was also found that under the optimal conditions (150 g/t Methyl isobutyl carbinol, 1500 g/t gas oil, and pH 4), the combustible recovery, yield reduction factor, and flotation efficiency index of coal reached to 67.79%, 0.056%, and 37%, respectively. Meanwhile, the FTIR analysis confirmed that the less adsorption of gas oil collector occurred in the presence of SDS (Sodium dodecyl sulfate) as frother due to the interaction of SDS and collectors.*

Keywords: *Flotation, Coal tailing, Zero prewetting time, Recovery, Optimization.*

1. INTRODUCTION

The average ash content is as high as 23.85% for power generation of commercial coal [1]. Approximately 6.5 million tons of coal tailing are produced in the Zarand coal washing plant, which are allocated in refuse ponds. These coal tailings remain untreated because of high cost and inefficient of processing. Disposal of tailings from the coal washing plant leads to loss of the recoverable amount of clean coal, significant huge area loss, and raising environmental pollution [2, 3, 4]. The utilization of energy sources is a necessary factor for the economic growth of a country [1]. Therefore, any method and suitable process for coal separation of the refuse tailings ponds could represent an important factor not only in producing energy but also in reducing environmental concerns. Several techniques have been reported for cleaning and utilizing the low rank/oxidized coals tails, such as gravity concentration techniques (Falcon concentrator, Knelson concentrator, Kelsey jigs, and Multi-Gravity separator (MGS)), and flotation. [2, 3, 5-12]. Among them, nowadays, the flotation process is widely used for the clean of coal tailings due to the limitations in the gravity concentration techniques such as the complex structure, the high cost of operation, and the dewatering problems owing to high water content of the final product [13]. Coal tailings are exposed to air and water and their surface characteristics are similar to low rank/oxidized coals. On the other hand, when the coal is oxidized, the hydrophilic oxygenated functional groups (such as carboxyl, phenol, and carbonyl functionalities) on the coal surface increases, and the floatable components (such as carbon and hydrogen - containing parts) of the coal decrease [14-17]. The hydrophobicity of coal surface reduces by increasing the hydrogen-bonded of oxygen functional groups with polar water molecules. Therefore, because of the formation of the stable hydrated film on the surface of oxidized coals, the conventional flotation process of low rank/oxidized coal is extremely difficult with common oily collectors, such as kerosene, diesel oil, and fuel oil [18, 19]. Many pretreatment methods were applied to enhance the floatability of low rank/oxidized coals, which includes, grinding [20], microwave [21, 22], thermal, [21-27] ultrasound, [28, 29] high-intensity

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conditioning, surface attrition, and direct mixing of the reagents with dry coal before wetting for flotation [30]. In addition to above methods, other techniques are also used to get a high yield or combustible matter recovery of difficult-to-float coal flotation. The surface modification method has been widely applied to enhance the recovery and hydrophobicity of low rank/oxidized coals with the effective chemical reagents [31-36]. The studies demonstrate that the compound surfactants/oxygenated functional groups and oily collectors, non-ionic oxygenated surfactants, the aliphatic alcohols, black oils, biodiesel, oxidized diesel oil, promoters, and blending of hydrocarbon and non-hydrocarbon collectors, such as copolymers, long- chain amine, fatty acid are applied to increase the floatability of oxidized coal. It is known that the low- rank coal/oxidized coals can be effectively floated using a series of non-ionic surfactants containing oxygenated and aromatic functional groups, i.e., tetrahydrofurfuryl esters (THF). The floatability of low rank/oxidized coals can also be improved by suitable bubble surface modification methods such as oily bubble flotation technology and nanobubble flotation column [37-40].

Although the effect of zero-conditioning time on the floatability of low rank/oxidized coals has been studied, no studies have been published into the optimization of zero prewetting time conditions for the effective flotation of oxidized coals and coal tailings. Also in the present study, the effect of compound zero prewetting time pretreatment and surfactant SDS (Sodium dodecyl sulfate) was investigated on the efficiency of different product ash percent. The objective of this work was to study the optimal conditions for the floatability of low rank/oxidized coals under zero prewetting time conditions. To achieve this purpose, the effect of the process parameters, i.e., pH, frother type, collector type on the flotation recovery of low rank/oxidized coal is discussed.

2. EXPERIMENTAL PROCEDURES

2.1. Materials

The present study was carried on coal tailings from the Zarand Coal Preparation Plant in Iran. The ash content of Zarand coal tailings in Iran is approximately 70%. According to the tailing layout, about 100 kg samples were collected from refuse ponds to obtain a representative sample. The samples from screening were combined and homogenized. A 26 kg sub- sample was split out for flotation experiments, using a riffle and then samples were passed from the 35 mesh (500 µm) screen to avoid the excessive grinding. A 1 Kg of particles remaining in the screen was ground using a laboratory rod mill for particle size reduction to less than 500 µm in diameter at 5 min. After each grinding period, samples were again passed from the 35 mesh (500 µm) screen. Material that does not pass through the screen has returned to the rod mill. The flotation tests were conducted in a Denver laboratory flotation machine with 1-l capacity using 183 g of the sample at a solids percentage of 10%. All operating parameters such as impeller speed, conditioning time, flotation time, and reagents dosage were kept constant during the experiments. The reagents used in the tests were gas oil (3000 g/t), Kerosene (3000 g/t), and a combination of gas oil (1500 g/t) and Kerosene (1500 g/t) as collector and methyl isobutyl carbinol (MIBC) (300 g/t), sodium dodecyl sulfate (SDS) (656 g/t) and a combination of MIBC (150 g/t) and SDS (328 g/t) as frother. In order to disperse of the collector into smaller droplets, collector, and water was added into the flotation cell and mixed at an impeller speed of 1600 rpm for 4 min first and then the impeller speed was kept constant at 1150 rpm and the sample of dry coal tailing was added into the flotation cell (zero prewetting time). The slurry mixture was conditioned for 90 s (collector stirring time). Afterward, MIBC used as a frothing agent was added, and the conditioning process was carried out for 30 s. Then flotation was started by introducing the air and the froth collected for 270 s. The flotation concentrate and tailing were filtered and then dried separately for each of the tests to obtain the recovery and ash content.

In order to evaluate the flotation results, the combustible matter recovery (E_C), flotation efficiency index (ε_c), yield reduction factor (YRF), index of collectivity (IC), index of selectivity (IS), and yield percentage of clean coal (Y) were used, which were calculated using Eqs. (1), (2), (3), (4), (5), and (6) respectively [47].

$$E_c(\%) = \frac{M_C(100-A_C)}{M_F(100-A_F)} \times 100 \quad (1)$$

$$\varepsilon_c(\%) = \frac{M_C(A_F-A_C)}{M_F A_F(100-A_F)} \times 100 \quad (2)$$

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$$YRF = \frac{M_F - M_C}{A_F - A_C} \quad (3)$$

$$IC = \frac{M_C}{M_T} \quad (4)$$

$$IS = A_C \frac{M_F}{M_C} \quad (5)$$

$$Y = \frac{A_T - A_F}{A_T - A_C} \times 100 \quad (6)$$

where M_C is the weight of the concentrate (%), A_C is the ash content of the concentrate (%), M_F is the weight of the feed (%), A_F is the ash content of the feed (%), and A_T is the ash content of the tailing (%). Fig.1 illustrate particle size distribution and ash content distribution of coal tailing.

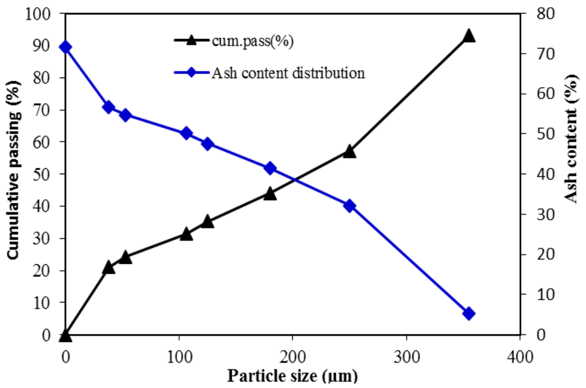

Fig. 1. Particle size distribution and ash content distribution of coal tailing.

2.2. FTIR Spectroscopy

Fourier transform infrared (FTIR) spectra of separated solids were studied to elucidate the effect of frother type on the recovery. The FTIR spectra of separated solids were obtained on a Bruker FTIR spectrometer using KBr discs in the spectral range of 4000-400 cm^{-1} and at a spectral resolution 1 of +4 cm^{-1}. The samples and KBr powder were mixed at a suitable blending ratio (by weight). The powder prepared using a mortar and pestle was soft and homogeneous, which was then pressed with a sheeter and transferred to the FTIR for measurements.

2.3. Experimental Design

The factorial design has been widely used to investigate the effect of each factor and interaction effects among different factors on each response in research. In this study, a 3^k factorial design was created to investigate the effect of collector type, frother type, and pH on the index of collectivity (IC), index of selectivity (IS), combustible matter recovery, flotation efficiency index, yield reduction factor and yield percentage of clean coal.

The main parameters are coded as -1, 0, and +1 which corresponds to the lowest level, the central level and the highest level respectively. The independent variables (frother type coded as A, collector type coded as B, and pH coded as C) and their levels are shown in Table 1. Table 2 shows the experimental conditions used in this study.

3. RESULTS AND DISCUSSIONS

3.1. Observed Responses

Fig. 2 displays the observed results for the combustible matter recovery and flotation efficiency index.

Table 1. Levels of variables in the experiments

Levels	Factors		
	A	B	C
-1	MIBC (M)	Kerosene (K)	4
0	MIBC+ SDS (M + S)	Kerosene + Gas oil (K + G)	6.5
+1	SDS (S)	Gas oil (G)	9

Table 2. Matrix considering the factorial design at three levels used in this work

Run	A	B	C	Run	A	B	C	Run	A	B	C
1	0	0	0	10	+1	-1	0	19	0	-1	0
2	+1	+1	0	11	0	+1	+1	20	+1	0	-1
3	+1	+1	+1	12	-1	+1	-1	21	+1	0	+1
4	0	-1	+1	13	-1	-1	+1	22	0	-1	-1
5	-1	-1	0	14	-1	0	-1	23	-1	+1	+1
6	0	0	+1	15	0	+1	0	24	-1	0	0
7	-1	0	+1	16	0	0	-1	25	-1	-1	-1
8	+1	0	0	17	+1	-1	+1	26	0	+1	-1
9	-1	+1	0	18	+1	+1	-1	27	+1	-1	-1

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Ac can be seen, the highest and lowest amount of combustible recovery occurred at the experiments 9 and 2 (68.92% and 51.28%), respectively. Experiments 9 and 2 are running with the same collector (gas oil) and pH (6.5) but with the opposite level of frothing agent (MIBC and SDS), respectively. This behavior indicates that the SDS as the frothing agent has the highest negative impact on the combustible matter recovery. In this study, the flotation efficiency index was used to indicate improvements in the coal tailing flotation. It is clear that the same trend with the combustible matter recovery is observed for the flotation efficiency index. However, its intensity values are different. It was also found that the experiments 2 and 14 was led to the minimum and maximum values of flotation efficiency index (0.22% and 0.38%), respectively. Experiment 14 was run in the lowest levels for the frothing agent and pH and the central levels for the type of collector. This behavior shows that the use of the lowest levels of pH (4) and a combination of gas oil and kerosene lead to a higher flotation efficiency index.

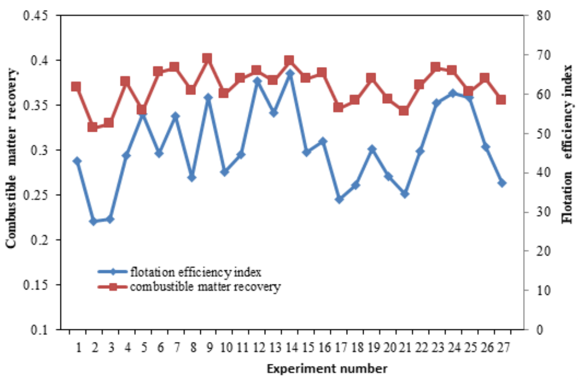

Fig. 2. Results of the experimental design for the combustible matter recovery and flotation efficiency index.

The IC was used to evaluate the collectivity of collectors. When the IC value of the collector is higher, the collectivity of the collector is better. The value of IC of different collectors is shown in The mean and standard deviation (Std) was evaluated for the IC value at each level of factor B (collector type). The results indicate that the mean IC value of a combination of gas oil and kerosene used as collectors is highest, therefore; the G+K is more suitable for coal tailing flotation. The results also show that the Std IC value of central level (K + G) of factor B has the lowest magnitude and so it is found that the collector of K + G is slightly affected by the change of other factors. In addition, the IC was employed to assess the collectivity of frother. The value of IC of different frothers is shown in Figure 4. The findings depict that the mean IC value of a combination of MIBC and SDSD used as frothers has the highest value. The results also indicate that the Std IC value of central level (MIBC + SDS) of factor A is the lowest, so this shows that the frothers of MIBC + SDS are less affected by changing other factors. Fig 4 shows that the mean IC value of SDS used as frother is fewer than MIBC.

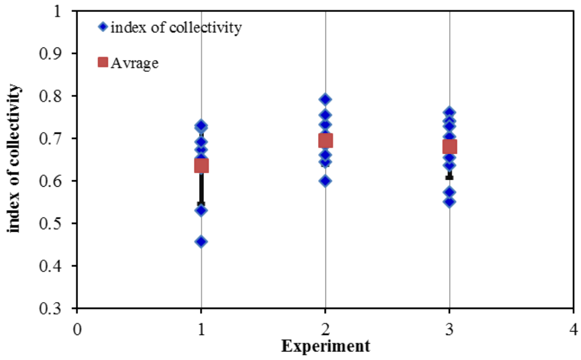

Fig. 3. Results of the experimental design for the index of collectivity (IC) of collectors

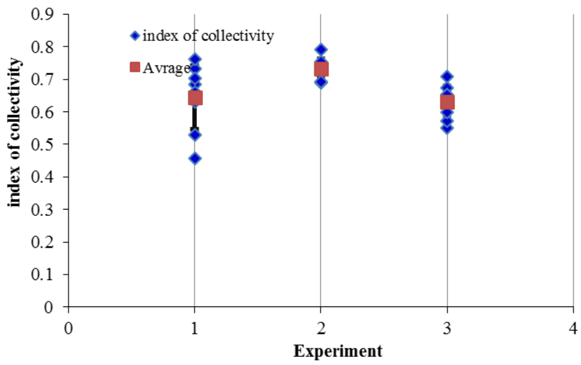

Fig. 4. Results of the experimental design for the

The index of selectivity (IS) was studied to compare the selectivity property of collectors. When the IS value of the collector is higher, the selectivity property of the collector is better. The value of IS for the different collectors is shown in Figure 5. The results show that the mean IC value of gas oil used as a collector is higher than the kerosene, therefor; the gas oil has more selectivity property for the flotation of coal tailings. Yield Reduction Factor (YRF) was used to evaluate the percent reduction in yield for each decrease in the

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ash content. It is clear that the coal washability is better when the Yield Reduction Factor is lower. According to Figure 6, experiments 2 and 25 are the maximum and minimum values of YRF (0.092 and 0.056), respectively.

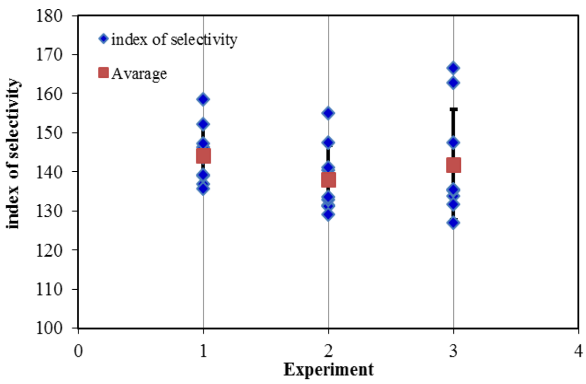

Fig. 5. Results of the experimental design for the index of collectivity (IS) of collectors

Experiment 25 is characterized by the highest level of frothers type and collector type, while the opposite occurs for experiment 2. These results show that the use of pH 4 leads to a better Yield Reduction Factor.

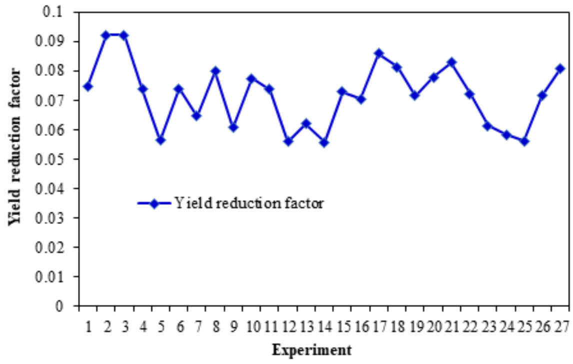

Fig. 6. Observed responses for the Yield Reduction Factor

3.2. Statistical Analysis

Analysis of variance (ANOVA) was conducted in order to check the statistically significant factors on the combustible recovery, YRF, and flotation efficiency index. The results of ANOVA are shown in Table 3. The results observed in Table 3 demonstrate that the type of frother and interaction between frother type and collector type were effective at 95% confidence level on all the three responses, and the pH was statistically significant on the flotation efficiency index and YRF.

Table 3. ANOVA for the combustible recovery, flotation efficiency index, and YRF of the factorial experimentation

Response	Source	Sum of Squares	df	Mean Square	F Value	p-value	
Combustible Recovery	Model	472.29	10	47.22	8.29	0.0001	significant
	A- frother type	323.96	2	161.98	28.45	< 0.0001	
	B-collector type	33.29	2	16.64	2.92	0.08	
	C-pH	6.07	2	3.038	0.53	0.59	
	AB	108.96	4	27.24	4.78	0.009	
	Residual	91.09	16	5.69			
	Cor Total	563.39	26				
flotation efficiency index	Model	0.05	10	0.005	50.18	< 0.0001	significant
	A- frother type	0.048	2	0.02	231.47	< 0.0001	
	B-collector type	0.00039	2	0.0001	1.85	0.18	
	C-pH	0.002	2	0.001	9.72	0.001	
	AB	0.001	4	0.0004	3.94	0.02	
	Residual	0.001	16	0.0001			
	Cor Total	0.05	26				
YRF	Model	0.002	10	0.0002	50.21	< 0.0001	significant
	A- frother type	0.002	2	0.001	229.38	< 0.0001	
	B-collector type	4.4E-05	2	2.24E-05	3.82	0.04	
	C-pH	0.0001	2	6.37E-05	10.89	0.001	
	AB	8.1E-05	4	2.03E-05	3.47	0.03	
	Residual	9.3E-05	16	5.85E-06			
	Cor Total	0.003	26				

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Effects of significant factors on all the three responses are shown in Figure 7. As shown in Fig. 7, when the frother type changed from the highest level to the lowest levels, the combustible recovery and flotation efficiency index increased, and the yield reduction factor reduced from 0.092 to 0.059. Meanwhile, when the pH value decreased from 9 to 4, the flotation efficiency index increased, while the yield reduction factor reduced.

3.3. Optimization Process

Finally, using these findings, the operating optimal point that combines combustible recovery, flotation efficiency index, and yield reduction factor could be predicted. Numerical optimization was performed using the desirability function approach in the Design Experts software to estimate the optimum levels of operating parameters with the aim of maximizing the combustible recovery and flotation efficiency index of coal and minimizing the yield reduction factor of coal. Fig. 8 shows the results of the process optimization. The optimal operating point attributed to experiment 12, in A= -1, B= +1, and C= -1 (frother type of MIBC, collector type of gas oil, and pH of 4), with 67.79 % and 0.37 %, 0.056 % for combustible recovery, flotation efficiency index, and yield reduction factor, respectively.

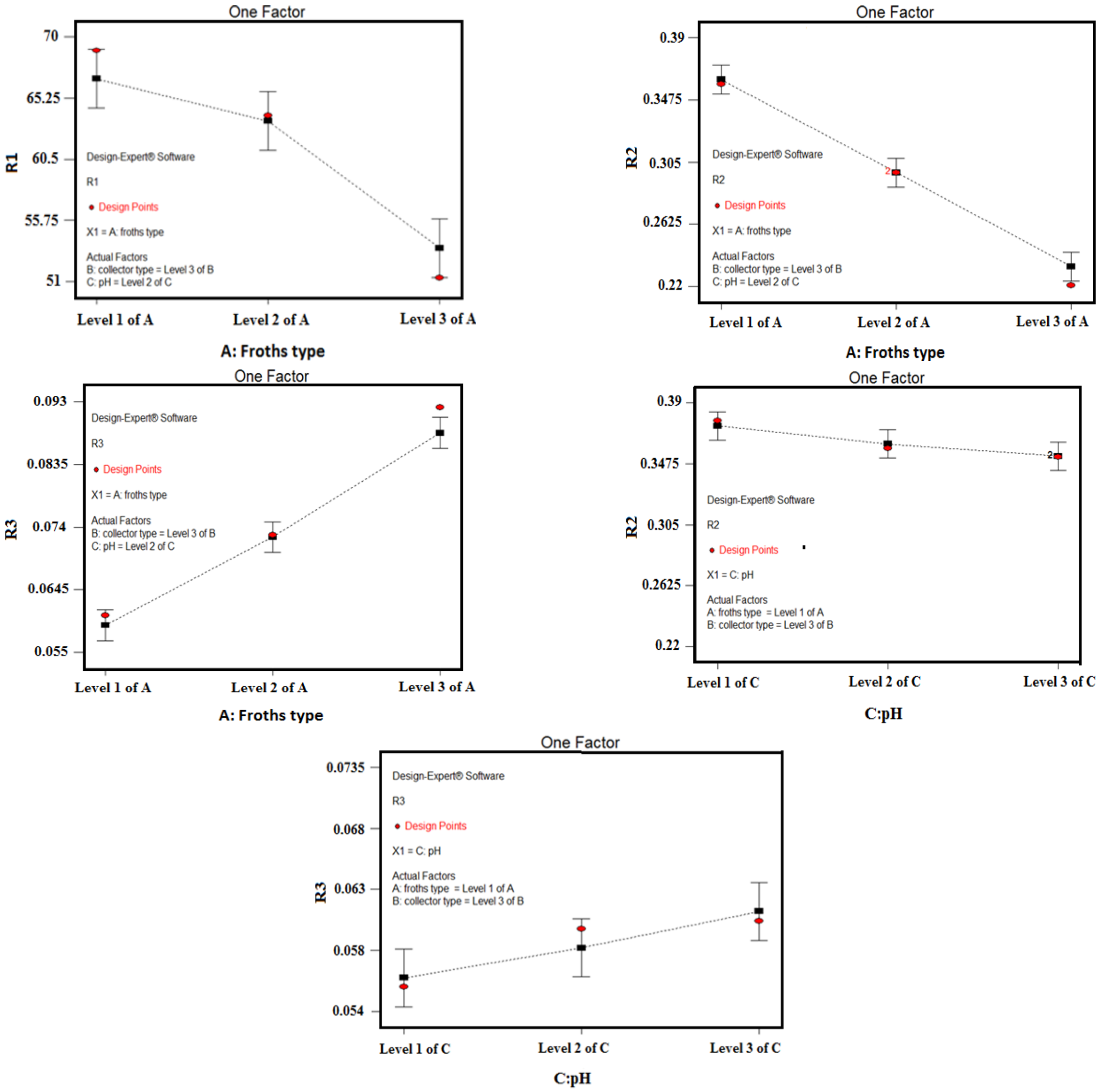

Fig. 7. The effect of frother type on the (a) combustible recovery (R1), (b) flotation efficiency index (R2), (c) yield reduction factor (R3), and the effect of pH on the (d) flotation efficiency index (R2) and (e) yield reduction factor (R3)

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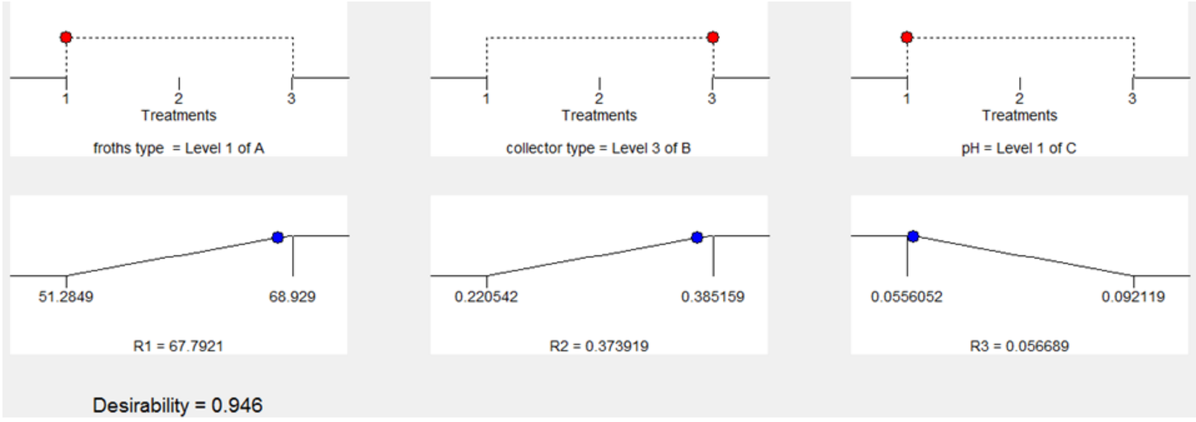

Fig. 8. The results of the optimization under the zero prewetting time pretreatment.

3.4. FTIR Studies

FTIR spectra of the highest and lowest combustible recovery (the experiments 9 and 2 respectively) are shown in Fig 9.

The adsorption peaks were attributed to OH (3429 cm^{-1}), -COOH (1622 cm^{-1}or 1595 cm^{-1}), R-H (1435 cm^{-1}) [41, 42, 43, 44].

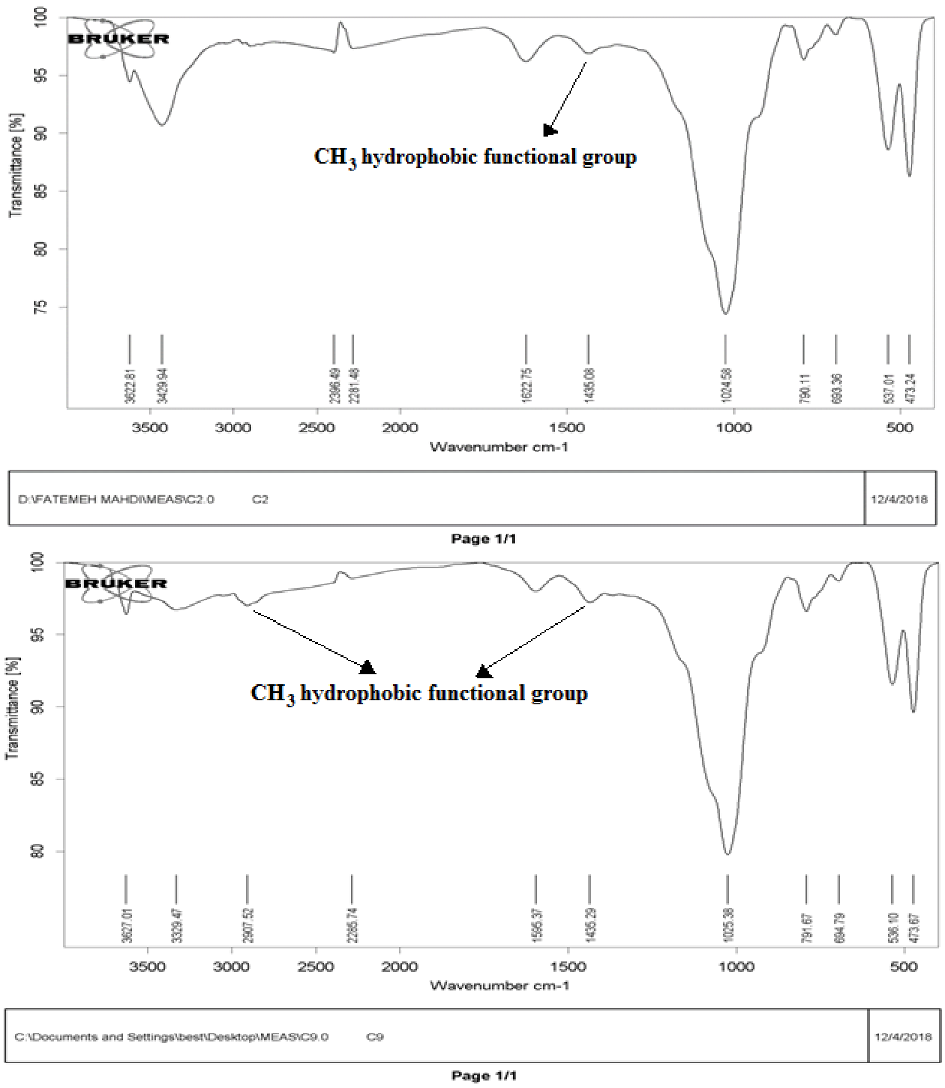

Fig. 9. The FTIR spectra of floated products in the experiments 2 and 9.

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It indicates that the adsorption peaks at 3429 cm^{-1} for the concentrate of experiment 2 were stronger and broader, which can be attributed to more hydrophilic of concentrate 2. In both FTIR spectra, the absorption band at 1435 cm^{-1} assigned to R-H or CH_3 of the hydrophobic functional group [42]. In addition, the absorption peaks at 1595 cm^{-1} or 1622 cm^{-1} represent the carboxyl group (-COOH), which is another hydrophobic functional group. The new absorption band found for concentrate 9 at 2907 cm^{-1} is attributed to the CH_3 hydrophobic functional group [45]. The results show that the concentrate of experiment 2 has fewer hydrophobic functional groups. This indicated that the coal sample under the conditioning process of experiment 2 has poor floatability. Similar observations were obtained by the flotation test. These peaks show that although a gas oil collector has been used for both experiments 9 and 2, the higher adsorption of the collector on the surface of feed in experiment 9 was obtained. The less adsorption of the collector in experiment 2 may be due to the interaction of SDS and gas oil collector. A band that appeared at 3622 cm^{-1} and 3627 cm^{-1} respectively at experiments 2 and 9 might be attributed to the moisture content in low- rank coal [46]. In addition, in order to study the influence of a combination of Gas oil and Kerosene as collectors on the surface of floated products, FTIR analysis of floated products at experiments 14 and 25 was recorded. Experiments 14 and 25 were run with the same frothing agent (MIBC) and pH (6.5) but with different levels of the collector (G + K and G), respectively.

Fig 10 indicates FTIR spectra on the floated products at experiments 14 and 25. The results show that the absorption bands of floated products at experiment 25 were similar to those in experiment 14. A new absorption band found for concentrate 25 at 3575 cm^{-1} for OH may be due to the less adsorption of the collector on the surface of the sample, which is in very good agreement with flotation results.

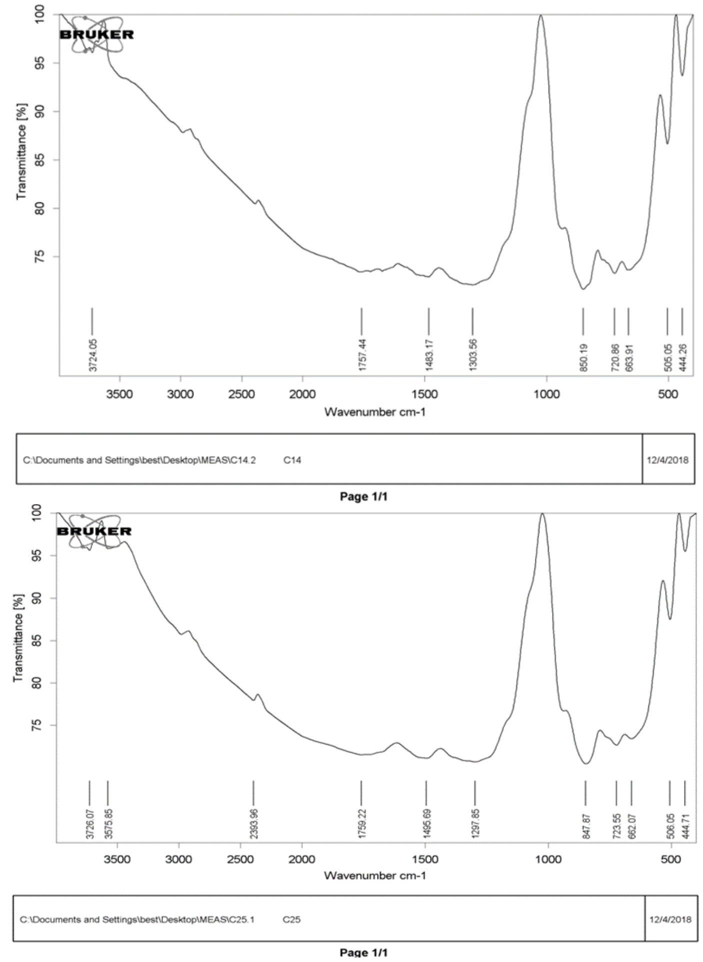

Fig. 10. The FTIR spectra of floated products in the experiments 14 and 25.

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4. CONCLUSIONS

This study investigated the rougher flotation stage of coal tailing under zero prewetting time pretreatment to reach an acceptable combustible recovery without paying attention to content ash. The results indicated that the frother type and interaction between frother type and collector type had significant effect in the 95% confidence level on the combustible recovery, flotation efficiency index, and yield reduction factor. The ANOVA also showed that the pH value was statistically significant on the flotation efficiency index and yield reduction factor. The optimization process demonstrated that the combustible recovery of 67.79%, the yield reduction factor of 0.056% and the flotation efficiency index of 37% could be obtained under prewetting time pretreatment with 150 g/t MIBC, and 1500 g/t gas oil at pH 4. It was also found that SDS as the frother agent had the highest negative impact on the combustible matter recovery. Meanwhile, the FTIR spectra results showed that less adsorption of the collector on the surface of the sample has occurred in the presence of SDS which may be due to the interaction of SDS and collectors. Moreover, the flotation efficiency index all experiments showed the same trend with the combustible matter recovery but its intensity values were different.

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