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# Effect of an acetonized pyrolysis oil recycled from spent-car tires on coal flotation performance

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**Abstract:** In this paper, an extended Historical Data (HD) design was applied for evaluating the effect of an acetonized pyrolysis oil (PO) produced by pyrolysis of spent-car tires in coal. Experimental and statistical analyses were applied for examining the influence of some operating variables such as concentration of diesel oil (0, 10, and 20 L/t), pine oil (0.55, 0.1, and 1 L/t), and the pyrolysis oil (0, 10, and 20 L/t) as well as solid content of pulp (5, 10, and 15% (w/w)) on the yield and ash content of final concentrate. Fourier Transform Infrared Spectroscopy (FTIR) measurements showed that PO contained hydroxyl, aldehyde, aliphatic, and aromatic compounds. Based on the results of Analysis of Variance (ANOVA), the main effect of all variables, except concentration of pine oil, on the flotation responses were found significant. Batch flotation experimental results indicated that using pyrolysis oil resulted in a 2% increase in ash content and a 35% decrease of the yield, through a nonlinear trend. The curved behavior of flotation measures was due to the possible competitive adsorption between PO and diesel oil and nonselective interaction between pyrolysis oil and other reagents. The negative effect of PO on coal flotation efficiency was also ascribed to the interaction between hydrophilic groups in PO structure and the oxide nature of non-combustible materials of coal particles.

*Keywords:* coal flotation, acetonized pyrolysis oil, ash rejection, experimental design, interaction effect

## 1. Introduction

Nowadays, there is quite a mixed variety of physical coal beneficiation methods including jigging, heavy media separation, and shaking table, though, it is well known that these techniques have not satisfactory performance in fine (ca. < 500 µm) coal processing (Jarkani et al., 2014; Khoshdast et al., 2014, 2019). To iron out such challenges, froth flotation has shown efficient performance in processing of fine particles. However, in order to obtain pleasant results in coal flotation, an optimal balance among various phases involved in flotation process should be provided (Kim et al., 1991). Providing hydrophobicity on the surface of coal particles by an appropriate collector is one of the technical goals of flotation process. One of the most common collectors increasing the orientation of coal particles to air bubbles are water-insoluble hydrocarbons which are generally non-polar oils like fuel-oil, kerosene, certain coal-tar distillates, and crude petroleum (Wojcik et al., 1990; Khoshdast, 2019). In general, the collectors are converted into droplets and dispersed in the droplet and these droplets collide with, adsorb to, and spread on the coal particles to increase their hydrophobicity (Polat et al., 2003).

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Today, majority of used collectors in coal flotation are petroleum-origin collectors, but the challenges such as environmental effects, depletion, and staggering costs of fossil fuels have led to many research works to explore alternative and renewable products and evaluating their collecting performance to decrease consumption of oil products in coal flotation (Yi et al., 2015a). For instance, Sönmez and Cebeci (2006) examined the effect of lubricating and classic oils on coal flotation. Results indicated that bright stock had the best flotation efficiency (about 10% lower ash content) with a higher combustible recovery (more than 4%) compared to conventional collectors. Xia et al. (2019) used lubricating oil as flotation collector and announced the enhanced recovery of clean low-rank. Besides, they examined the involved mechanisms of flotation process by conducting comprehensive investigations on characterization of such oils. Zhen et al. (2019) used plasma oxidation technique to examine the influence of oxidized diesel oil on the flotation of the low-rank coal. Results showed that oxidation of the low-rank coal wetted by the diesel oil leads to decreasing the contact angle from 34.5° and 23.7°. Afterward, Yang et al. (2021) investigated the effect of waste motor oils on the flotation performance of low-rank coal slime and obtained a total yield of 77.29% and an ash content of 8.97% at the dosage of 5 kg/t, which compared to diesel oil, it was an impressive development. Xia and co-workers (2018) applied a mixture of hydrocarbon oil and candle soot as a novel flotation collector for improving the coal flotation. They reported that soot adsorbed on the surface of coal particles and enhanced the flotation efficiency (about 8% higher recovery and 2% lower ash content) and the hydrophobicity of particles, particularly in the case of low-rank and coking coals. In another study, Xie et al. (2010) investigated flotation of coal slime through a depressing/floating procedure and using wash oil, one fraction of coal tar, as a coal collector for overcoming demerits of conventional collectors. Results indicated that by applying this novel technology, the ash content was reduced by 0.17% and the yield was enhanced by 5.3%.

Besides, Li et al. (2020) examined flotation of a low-rank coal using a novel coal tar-based (CTB) collector. Results indicated 21%-34% higher combustible matter recovery of collector compared to the diesel oil. Likewise, the CTB collector showed a favourable flotation performance by saving 1500 g/t dosage than diesel oil. Recently, Xia et al. (2021) examined the modification of low-rank coal floatability by a compound collector consisting of coal tar and diesel. Surface analyses showed that this improvement was due to enhanced hydrophobicity of the surface of coal particles interacted by coal tar compounds. Through a novel study, Fazaelipoor and co-workers (2010) used a mixture of rhamnolipid biosurfactants and diesel oil for coal flotation. Results showed that this biosurfactant could decrease the diesel oil consumption while enhancing the coal separation by 2.5 %. Yi et al. (2015a,b) investigated possibility of using waste cooking oil as a biocollector. It was reported that compared to diesel oil, waste cooking oil could provide suitable flotation efficiency with a 13 times lower consumption and can be considered as a suitable alternative of diesel oils. Some other investigators examined the usage of other types of waste food oils. For instance, the recovery of coal flotation was enhanced by 10% using gaseous pyrolysis products of the waste cooking oil. Xu et al. (2019) also reported this improvement using waste colza oil for low-rank coals. Cheng et al. (2020) used a collector produced from waste hot-pot oil in lignite flotation and showed that their novel collector provide the separation efficiency and ash content of 3.04% higher and 1.14% lower than that of the conventional collector of kerosene. Yang and co-workers (2020) announced enhanced efficiency of coal fly ash using fried oil as a collector and for carbon depressant. Another study investigated the use of waste cooking oil for enhanced recovery of unburned carbon (higher than 84%) from coal fly ash (Yang et al., 2021). Recently, Cheng et al. (2024) developed a novel method for the green utilization of waste fried oil to prepare different types of reagents following a simple multi step process including waste fried oil treatment, orange peel extract preparation, saponification, demoulding and drying. Results showed that the produced reagents are of significantly low toxicity compared to reagents with petroluem origin.

An efficient collector for separation of coal substance from kaolinite is polyaluminum chloride (PAC) (Liang et al., 2016, 2019). Results indicated that PAC did not aggregate coal, while floating less ash material. They revealed that presence of 2-ethylhexanol, particularly in a mixture with diesel oil, could enhance flotation performance by increasing recovery of combustible matter and flotation efficiency index from 43.42% and 29.52% up to 89.27% and 59.91%, respectively. Several researchers applied a quite a mixed variety of polymeric and surface active reagents, either discretely or in mixed composition, for coal flotation such as dodecane (Wang et al., 2019; Zhang et al., 2020), gemini surfactant

(Zhu et al., 2020), non-ionic surfactant triton X-100 (Zheng et al., 2021a,b), anionic polyacrylamide (Xia et al., 2021), polyvinylpyrrolidone (Wang et al., 2021), dodecyl ethoxyl ethers (Li et al., 2019), and polystyrene (Liao et al., 2021). Diverse parameters such as the number of ethoxyl functional group in their structure, the length of hydrocarbon chain and their consumption dosage affect the regent performance. Li et al. (2022) provided a deep literature review on exploration of the effect of different types of polymeric reagents as well as their interaction mechanisms in coal flotation.

In this study, effect of acetonized pyrolysis oil recycled from spent-car tires on coal flotation performance was investigated. An extended experimental design was used to evaluate response of a bituminous coal to the ash rejection and total yield flotation with this recycled oil. According to the individual and interaction effect plots, effect of some parameters such as dosages of pine, diesel and pyrolysis oils as well as pulp solid content were investigated. Besides, instrumental analysis was used for examining the potential mechanism involved in final flotation response.

## 2. Materials and methods

### 2.1. Coal sample preparation and characterization

Sampling campaign was executed for three-days long with two-hour intervals in each working shift from Zarand Coal Washing Plant (Zarand, Iran). To eliminate the influence of flotation reagents on samples, the sampling process was performed from the pulp stream, just before entering to the conditioning tank. Afterwards, the samples were filtered, dried in an oven at 60°C and blended for providing a bulk sample. A bulk sample with the required weight was prepared by riffling procedure (Khoshdast et al., 2012). Based on ASTM D 3174-73 standard, a typical ash analysis technique was used to measure the combustible content of the coal sample. A 900 gram of the bulk sample was used for determining the particle size distribution of coal particles using a standard wet sieve analysis technique. Besides, X-ray powder diffraction (XRD, Philips, X'pert-MPD system, The Netherlands) and the X-ray fluorescence (XRF, Philips, Magix-601, The Netherlands) techniques were applied to determine the mineralogical and chemical compositions of the sample, respectively (Khoshdast and Shojaei, 2012). Representative samples were also prepared and homogenized for flotation experiments.

### 2.2. Production and characterization of pyrolysis oil

In this study, the developed technique by Arjomand (2020) was applied for producing the used pyrolysis oil. For providing a better comprehension, the involved stages of recycling process are illustrated in Fig. 1. Rested on the claim of innovator, the developed pyrolysis process was based on a closed loop path such that all gaseous vapours emitted from pyrolysis process could be completely gathered and recycled as liquid water and a solid powder. Thus, the process was claimed to be green and totally compatible with the environment. Afterward, Fourier-transform infrared (FTIR, Bruker tensor 27, Berlin, Germany) with a RT-DLATGS detector was used for analyzing the final dark brown oily product and detecting its functional groups.

### 2.3. Operating variables and experimental design

Based on the suggestions of plant research and development division and for optimization purposes, effective operating variables were opted, and a six-month monitoring period was considered for determining levels of each considered parameter. Table 1 lists the parameters and their considered levels in experimental design. Technique of Historical Data (HD) experimental design was applied for evaluating impact and interactions of each parameter. To assess nonlinear effects of each variable, three levels were selected for the HD design. The considered process responses of this studies included total yield (%) and the ash content (%) of the concentrate. The practical results of experimental design are shown in Table 2.

### 2.4. Flotation experiments and calculations

Flotation experiments were performed in a standard 2 L (D-12) Denver® flotation machine. In order to fix the pulp level, a constant amount of water was added to the cell. A natural pH of 7±0.2 was consider-

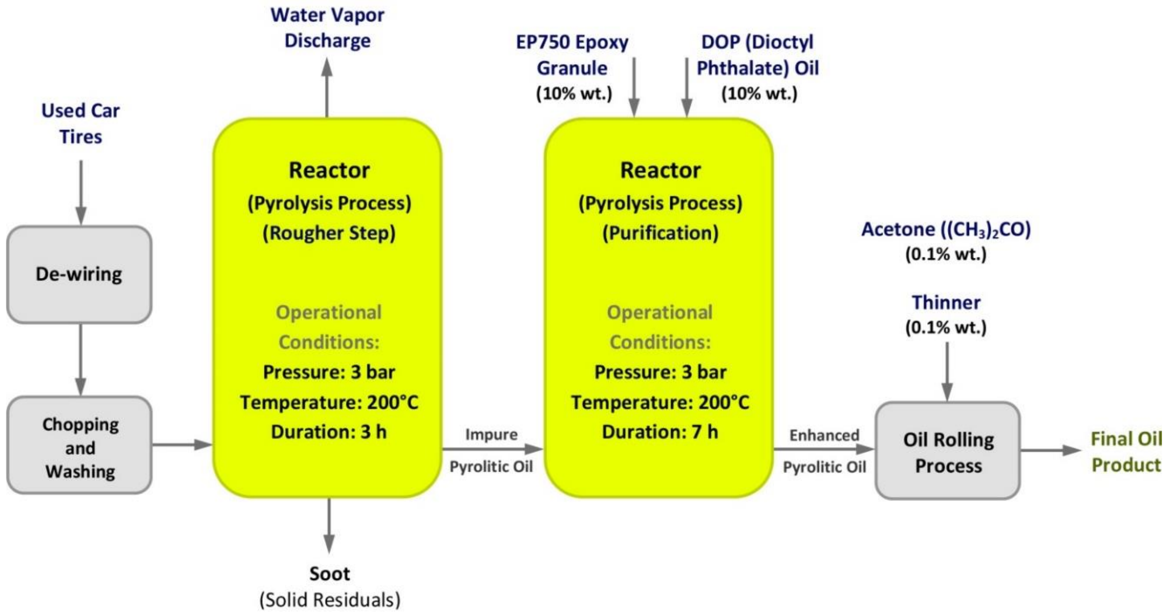


Fig. 1. Illustration of sub-processes involved in recycling pyrolysis oil from used-car tires

Table 1. Operating factors and their studied levels in the experimental design

| Factor | Name | Units | Low Actual | High Actual | Mid Level | Std. Dev. |
|---|---|---|---|---|---|---|
| A | Solid content | (%, w/w) | 5 | 15 | 10 | 4.12 |
| B | Pine oil conc. | (L/t) | 0.1 | 1 | 0.55 | 0.36 |
| C | Diesel oil conc. | (L/t) | 0 | 20 | 10 | 8.21 |
| D | Pyrolysis oil conc. | (L/t) | 0 | 20 | 10 | 6.03 |

ed for all experiments; besides, pine oil and diesel oil were used as a frother and collector, respectively. In order to make a pulp with the desired solid content (Table 2) in each experiment, first, a suitable amount of coal material was blended with 1 L of tap water, afterward, for complete soaking of all coal particles, it was agitated at impeller speed of 1000 rpm for 5 min. Then, water was added to the system and cell was filled to a set level. Subsequently, a suitable and required dosage of flotation reagents and pyrolysis oil were added to the system and were conditioned for another 5 min (Khoshdast and Sam, 2012). Eventually, the cell was aerated and for 3 min long, the formed forth was conveyed to the concentrate vessel. For each experiment, the collected concentrates and tailings were weighed and dried in an oven at 50°C for 4 h. Samples were then sent for ash analysis using the standard procedure mentioned in section 2.1 (Khoshdast et al., 2011a). The ash content and yield of concentrate were considered as metallurgical responses of coal flotation. These metallurgical measures are the most common ones used in Iran to evaluate the flotation performance of coal materials. However, other criteria such as coal recovery and separation efficiency can be further considered (Cheng et al., 2022). Equation (1) was used for calculating the experimental total yield ($Y$, %) (Gholami and Khoshdast, 2021):

$$Y(\%) = \frac{a_t - a_f}{a_t - a_c} \times 100 \quad (1)$$

where $a_f$, $a_c$, and $a_t$ are the ash contents of feed, concentrate, and tailings in percentage, respectively.

### 2.5. Supplementary FTIR analysis

Another two FTIR analyses were conducted for investigating the possible interaction of pyrolysis oil with the surface of coal particles. Firstly, deionized water was used for washing the raw coal sample and after drying, it was put into a vacuum bag. Besides, pyrolysis oil at a concentration of 20 L/t and 100 g of coal were added to the flotation cell containing deionized water and gently agitated for 30 min. Then, the coal was filtered and dried (Mirshekari et al., 2022). Finally, both raw and exposed coal samples were sent for the FTIR analysis.

Table 2. The structure and practical results of experimental design

| | Operating factors | | | | Responses | |
|---|---|---|---|---|---|---|
| Run | Solid content | Pine oil dose | Diesel oil dose | Pyrolysis oil dose | Ash | Yield |
| 1 | 15 | 0.1 | 20 | 0 | 12.8 | 65.3 |
| 2 | 10 | 0.55 | 0 | 0 | 9.3 | 7.0 |
| 3 | 15 | 1 | 20 | 0 | 14.5 | 66.2 |
| 4 | 5 | 1 | 10 | 10 | 13.1 | 60.6 |
| 5 | 5 | 0.55 | 0 | 0 | 10.0 | 47.2 |
| 6 | 5 | 0.1 | 0 | 0 | 6.6 | 15.0 |
| 7 | 10 | 0.1 | 10 | 0 | 14.9 | 67.0 |
| 8 | 10 | 1 | 0 | 10 | 14.4 | 58.5 |
| 9 | 5 | 1 | 0 | 10 | 17.4 | 72.4 |
| 10 | 5 | 0.55 | 20 | 0 | 14.7 | 64.6 |
| 11 | 5 | 0.1 | 0 | 20 | 14.8 | 63.2 |
| 12 | 10 | 0.55 | 20 | 10 | 13.9 | 58.4 |
| 13 | 5 | 0.1 | 20 | 0 | 14.7 | 65.6 |
| 14 | 5 | 0.1 | 10 | 0 | 13.6 | 63.6 |
| 15 | 15 | 0.55 | 20 | 0 | 13.8 | 62.7 |
| 16 | 15 | 0.1 | 0 | 10 | 12.8 | 49.9 |
| 17 | 10 | 0.55 | 10 | 10 | 13.8 | 56.4 |
| 18 | 15 | 1 | 20 | 10 | 11.9 | 45.8 |
| 19 | 15 | 1 | 0 | 0 | 10.3 | 43.2 |
| 20 | 5 | 1 | 0 | 0 | 10.4 | 42.7 |
| 21 | 10 | 1 | 0 | 0 | 12.4 | 45.5 |
| 22 | 5 | 0.55 | 10 | 0 | 7.27 | 64.5 |
| 23 | 15 | 0.1 | 10 | 0 | 12.8 | 66.3 |
| 24 | 10 | 0.1 | 20 | 0 | 14.5 | 68.1 |
| 25 | 15 | 0.1 | 20 | 10 | 12.7 | 46.8 |
| 26 | 5 | 1 | 20 | 10 | 17.2 | 13.8 |
| 27 | 5 | 1 | 20 | 0 | 12.1 | 67.4 |
| 28 | 15 | 1 | 10 | 10 | 15.0 | 62.9 |
| 29 | 10 | 0.1 | 0 | 10 | 10.2 | 35.3 |
| 30 | 10 | 0.1 | 20 | 10 | 12.1 | 43.2 |
| 31 | 15 | 0.55 | 0 | 10 | 14.0 | 53.9 |
| 32 | 15 | 0.55 | 0 | 0 | 11.6 | 18.2 |
| 33 | 5 | 0.1 | 0 | 10 | 16.5 | 57.0 |
| 34 | 15 | 0.55 | 10 | 10 | 14.8 | 62.0 |
| 35 | 10 | 0.1 | 0 | 0 | 8.3 | 10.3 |
| 36 | 5 | 0.1 | 20 | 10 | 12.6 | 61.4 |
| 37 | 10 | 1 | 10 | 10 | 12.1 | 46.5 |
| 38 | 15 | 0.1 | 0 | 0 | 11.1 | 28.4 |
| 39 | 10 | 1 | 10 | 0 | 12.3 | 66.9 |
| 40 | 10 | 0.55 | 20 | 0 | 13.7 | 67.7 |
| 41 | 15 | 0.55 | 10 | 0 | 12.4 | 62.7 |
| 42 | 5 | 1 | 10 | 0 | 11.5 | 64.3 |
| 43 | 5 | 0.55 | 10 | 10 | 18.6 | 82.5 |
| 44 | 10 | 1 | 20 | 0 | 12.8 | 67.3 |
| 45 | 15 | 0.55 | 20 | 0 | 13.8 | 62.7 |
| 46 | 5 | 1 | 10 | 20 | 14.8 | 75.7 |
| 47 | 10 | 0.55 | 10 | 0 | 13.5 | 63.9 |
| 48 | 15 | 1 | 0 | 10 | 14.6 | 60.4 |
| 49 | 15 | 0.55 | 10 | 20 | 14.8 | 75.7 |
| 50 | 10 | 0.55 | 0 | 10 | 13.6 | 52.5 |
| 51 | 15 | 1 | 10 | 0 | 12.8 | 64.4 |
| 52 | 5 | 0.55 | 0 | 10 | 13.0 | 62.8 |
| 53 | 10 | 0.55 | 0 | 0 | 9.6 | 6.8 |

## 3. Results and discussion

### 3.1. Characterization of coal sample and pyrolysis oil

Ash analysis showed that the bituminous coal sample consisted of 31.95 wt% non-combustible material. Based on the size distribution of ash materials which is shown in Fig. 2, the content of ash material increases as the size of coal particles decreased. The main ash content (63.6%) is situated in the finest size fraction (< 150 μm). Results of the XRF analysis (Table 3) showed that Al and Si oxides are the main components of the non-combustible portion. Additionally, according to the XRD analysis, illite

($Al_2H_2KO_{12}Si_3$), a group of closely-related non-expanding clay minerals, and quartz ($SiO_2$) with a minority of calcite ($CaCo_3$) and hematite ($Fe_2O_3$) were the main components of the ash materials.

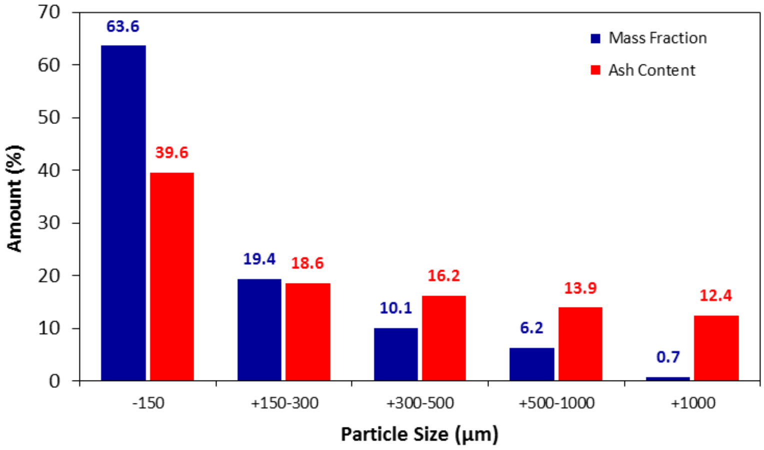


Fig. 2. Size-by-size retaining mass distribution and corresponding ash contents of the coal feed

Table 3. Chemical composition of non-combustible portion of the studied coal sample

| Component | $SiO_2$ | $Al_2O_3$ | $Fe_2O_3$ | CaO | MgO | $SO_3$ | $Na_2O$ | $K_2O$ | Loss |
|---|---|---|---|---|---|---|---|---|---|
| Portion (%) | 52.1 | 25.9 | 5.3 | 3.8 | 2.8 | 2.3 | 2.8 | 4.3 | 0.7 |

As shown in Fig. 3a, FTIR spectrum shows that the analyzed pyrolysis oil is not a simple chemical substance. A broad absorption band in the range of between 3650 and 3250 $cm^{-1}$, indicating hydrogen bond. This band which followed by the presence of spectra at frequencies of 1600–1300, 1200–1000, and 800–600 $cm^{-1}$ confirms the existence of hydroxyl (–OH) (Nandiyanto et al., 2019). Absorption band for long-chain linear aliphatic compounds is identified at 2935 and 2860 $cm^{-1}$ which is followed by peaks at between 1470 and 720 $cm^{-1}$. In identifying the double bond region (1500-2000 $cm^{-1}$), below 1700 $cm^{-1}$ indicates amides or carboxylates functional group. Also, strong intensity at between 1650 and 1600 $cm^{-1}$ inform double bonds or aromatic compounds. Moreover, spectra between 1615 and 1495 $cm^{-1}$ are responsible for aromatic rings. Two sets of absorption bands around 1600 and 1500 $cm^{-1}$ which are followed by the existence of weak to moderate absorption in the area of between 3150 and 3000 $cm^{-1}$ (for C–H stretching) confirm the existence of aromatic rings (Nandiyanto et al., 2019; Mirshekari et al., 2023).

### 3.2. Statistical analysis of results

In order to analyse the effect of operational parameters on process responses, firstly, it is necessary to develop a parametric model that can accurately forecast the intended response in the operating area, i.e., inside the low to high intended range for parameters (Boveiri et al., 2019). For modelling experimental data, the Design Expert v.7.0 software (Demo version 7.0.0, from Stat-Ease Inc., Minneapolis, MN, USA) was used. Secondly, after development of the initial model by the software,

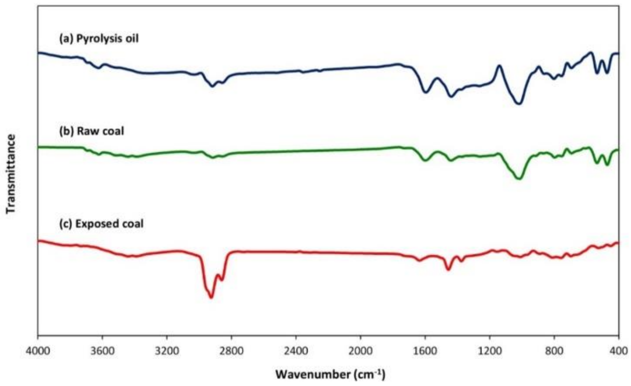


Fig. 3. Demonstration of the FTIR spectra of (a) pyrolysis oil, (b) raw coal, and (c) exposed coal

the model parameters were examined for detecting abnormal data and user then optimized the model for achieving the best fitting results. The result of these measures for the data obtained in flotation experiments was the development of nonlinear models for all process responses as below:

$$Ash\ content = 12.954 + 0.214A + 0.275B - 0.552C + 1.069D - 0.002AB - 0.273AC - 0.437BC + 0.608BD - 2.160CD \tag{2}$$

$$Yield = 58.698 - 4.161A - 0.761B - 6.037 - 13.357D - 4.179AD - 6.454BC - 5.111BD - 26.433CD + 6.735A^2 + 3.650B^2 - 16.741C^2 - 16.011D^2 \tag{3}$$

where factors are in coded form. Table 4 shows the validation parameters for the developed models. According to the Table 4 and due to high value of Fisher's F-test and marginal probability value ($p$ model < 0.0001), the suggested prediction models are all significant. One of the appropriate tools for assessing the significance of the prediction model is the residuals normal probability plot (Shami et al, 2021). Based on the relatively uniform normal probability plots of all responses (Figure 4), the normality assumptions and independence of the residuals during the statistical analyses can be confirmed. Additionally, the significance of the prediction models can be indicated by the high values of the adjusted correlation coefficients. Based on the reasonably high values of Pred $R^2$ and reasonable agreement with the Adj $R^2$ values, the ability of the model to explain variability in predicting new observations with acceptable accuracy is shown (Gholami et al., 2021). This is also evident from the plots showing the predicted values versus experimental data in Figure 5. Another statistical tool for showing the signal-to-noise ratio is Adeq precision with a value greater than 4 (Mahmoodabadi et al., 2019). In this study, the ratios were 15.30 and 14.99 for yield and ash content, respectively. These values show an appropriate signal so that the models can be applied for developing and predicting the design space.

Table 4. Validation parameters showing the significance of models for flotation responses

| Model | F Value | $p$-value | $R^2$ (%) | Adj $R^2$ (%) | Pred $R^2$ (%) | Adeq Precision |
|---|---|---|---|---|---|---|
| Ash content | 19.79 | < 0.0001 | 92.91 | 91.48 | 88.97 | 14.99 |
| Total yield | 18.42 | < 0.0001 | 93.45 | 91.92 | 89.32 | 15.30 |

Equations (2) and (3) were used to assess the significance of operating variables on process responses. Tables 5 and 6 show the ANOVA results within a confidence interval of 95%. As shown in Tables 5 and 6, except for pine oil dosage, the effects of other operational variables on process responses are statistically meaningful due to p-values less than 0.05. Interestingly, although the individual effect of frother concentration is not significant, there is a meaningful interaction between this factor with diesel oil and pyrolysis oil, i.e., BC and BD for ash content and BC for yield. Moreover, there is a highly significant interactive effect between diesel oil and pyrolysis oil for both flotation responses, i.e., DC. The insignificant interactions in both ANOVA tables are those that had a positive effect in the accuracy of prediction models. However, they have not interpreted in the following section.

### 3.3. Interpretation of main and interaction effects

One of the effective tools for assessing the influence of each individual parameter on the process response is main effect plots. In these plots, the response is drawn for considered levels of parameters in the experimental design (Khoshdast et al., 2021). In order to calculate the response values for variables based on their experimental levels, the developed prediction model (Eqs. (2) and (3)) were used keeping other parameters at their mid-levels. Figures 6 and 7 illustrate the main effects plots of different operating variables for ash content and yield, respectively. As shown in Figure 6, the effects of solid content and pine oil concentration on ash content in concentrate follow a gentle linear trend indicating that these effects are not considered as predicted in the ANOVA (Table 7).

By increasing the concentration of diesel oil as the coal collector, the ash content in the concentrate decreases, clearly due to improved selective floatability of coal particles, which leads to the improved performance of coal flotation. In contrast, pyrolysis oil increases the ash content through a rising trend. As shown in FTIR spectrum (Fig. 3), the structure of pyrolysis oil consists of several hydroxyl groups. These hydrophilic functional groups can interact with the surface of non-combustible material, which has an oxide nature (Table 3) and improve their floatability (Fozooni et al., 2017). This phenomenon indicates that the activity of hydrophilic groups in the structure of pyrolysis oil and their tendency to interact with the hydrophilic surface of non-combustible material is greater than the Bond energy (most likely van der Waals) between the hydrocarbon group and the coal surface. As a result, the proportion of particles containing non-combustible materials in the froth and concentrate increases.

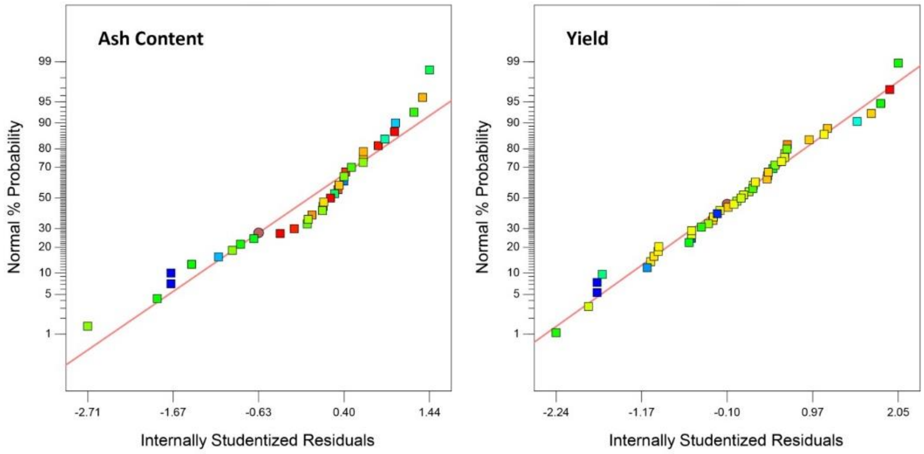


Fig. 4. Normal plots of the residuals for flotation responses of ash content and total yield

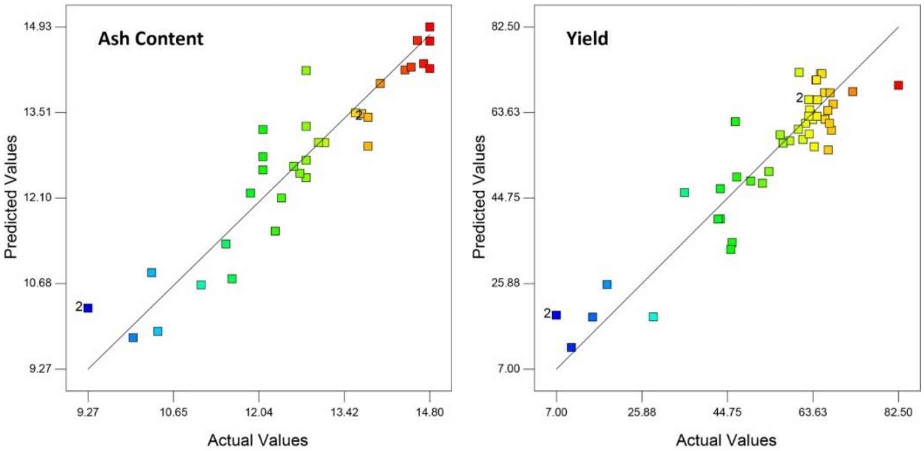


Fig. 5. Predicted vs. actual plots for flotation responses of ash content and total yield

Table 5. Analysis of variance results for ash content of the concentrate

| Source | Sum of Squares | df | Mean Square | F Value | $p$-value (Prob > F) |
|---|---|---|---|---|---|
| Model | 73.18056 | 9 | 8.131174 | 19.79871 | < 0.0001 |
| A-Solid content | 0.965129 | 1 | 0.965129 | 2.350006 | 0.1378 |
| B-Pine oil conc. | 1.141546 | 1 | 1.141546 | 2.779566 | 0.1080 |
| C-Diesel oil conc. | 3.468304 | 1 | 3.468304 | 8.44502 | 0.0076 |
| D-Pyrolysis oil conc. | 14.64307 | 1 | 14.64307 | 35.65462 | < 0.0001 |
| AB | 3.36E-05 | 1 | 3.36E-05 | 8.19E-05 | 0.9929 |
| AC | 1.226423 | 1 | 1.226423 | 2.986235 | 0.0963 |
| BC | 2.480259 | 1 | 2.480259 | 6.039216 | 0.0213 |
| BD | 2.959795 | 1 | 2.959795 | 7.206844 | 0.0127 |
| CD | 33.07254 | 1 | 33.07254 | 80.52877 | < 0.0001 |
| Residual | 10.2673 | 25 | 0.410692 | | |
| Lack of fit | 10.2673 | 23 | 0.446405 | | |
| Cor Total | 83.44787 | 34 | | | |

Table 6. Analysis of variance results for total yield to the concentrate

| Source | Sum of Squares | df | Mean Square | F Value | *p*-value (Prob > F) |
|---|---|---|---|---|---|
| Model | 99938.16 | 40 | 2498.454 | 43.62388 | < 0.0001 |
| A-Solid content | 298.7623 | 1 | 298.7623 | 3.278710 | 0.0421 |
| B-Pine oil conc. | 6.526622 | 1 | 6.526622 | 0.112239 | 0.7396 |
| C-Diesel oil conc. | 370.753 | 1 | 370.753 | 6.375889 | 0.0163 |
| D-Pyrolysis oil conc. | 316.4313 | 1 | 316.4313 | 5.441711 | 0.0255 |
| AD | 128.1794 | 1 | 128.1794 | 2.204319 | 0.1466 |
| BC | 847.386 | 1 | 847.386 | 14.57261 | 0.0005 |
| BD | 181.1436 | 1 | 181.1436 | 3.11515 | 0.0863 |
| CD | 4565.822 | 1 | 4565.822 | 78.51905 | < 0.0001 |
| A2 | 468.7169 | 1 | 468.7169 | 8.060586 | 0.0075 |
| B2 | 133.5514 | 1 | 133.5514 | 2.296702 | 0.1386 |
| C2 | 2517.698 | 1 | 2517.698 | 43.29719 | < 0.0001 |
| D2 | 433.3234 | 1 | 433.3234 | 7.451921 | 0.0099 |
| Residual | 2035.223 | 35 | 58.14923 | | |
| Lack of fit | 2035.223 | 33 | 61.67342 | | |
| Cor Total | 14888.22 | 47 | | | |

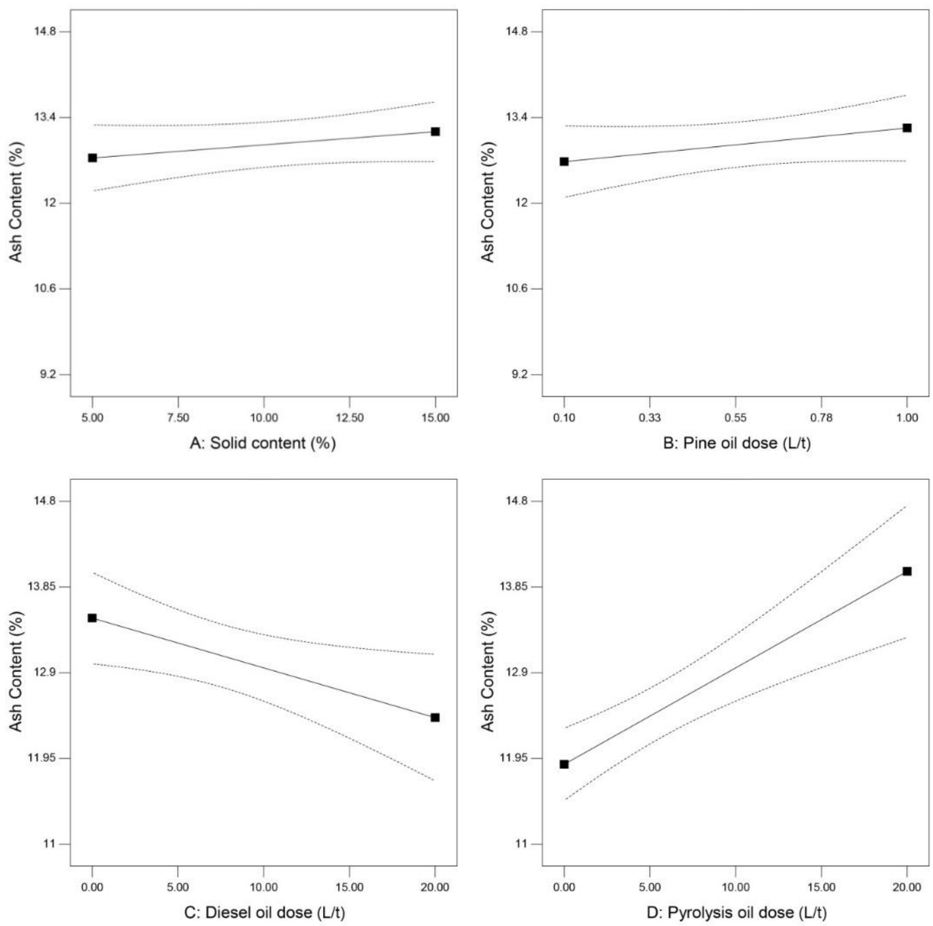


Fig. 6. Main plots show the effect of operating variables on ash content

As can be seen in the main effect plots of yield in Fig. 7, there is a nonlinear relationship between the yield and solid content. As the pulp solid percentage increases, the mass recovery to the concentrate remarkably drops to a plateau and then grows again toward the upper limits of solid content. As the solid content increases, the consequent increased turbulent condition in pulp interrupt with the efficient particle/bubble collision and thus, bubbles are forced to release their load. Therefore, the flotation rate of particles toward the froth and next, to the concentrate reduces. At higher levels of solid content, the excessive turbulent regime in pulp may be responsible for the increased total yield to the concentrate due to mechanical entrainment (Paryad et al., 2017; Zahab-Nazouri et al., 2022; Khoshdast et al., 2023). Noteworthy that during the experiments performed at a high percentage of solid, a large amount of turbulence was observed on the surface of the froth. Similar to the ash content, the effect of frother concentration on yield is not significant as predicated in ANOVA. The effect of the collector on yield is also nonlinear such that the yield increases up to a middle level of diesel concentration due to the improved hydrophobicity of coal particles, and then tends to decrease likely because of the unwanted flotation of ashy coal particles with less liberation degree. The effect of pyrolysis oil on yield is relatively negligible up to a concentration of about 10 g/L where the yield significantly drops by around 40%. Comparing the main effect plots for PO dosage on ash content and yield, PO may act as an activator for ash material and a depressant for coal material. To explore the proposed mechanism, the surface of the coal sample before and after exposure to PO was studied by FT-IR analysis.

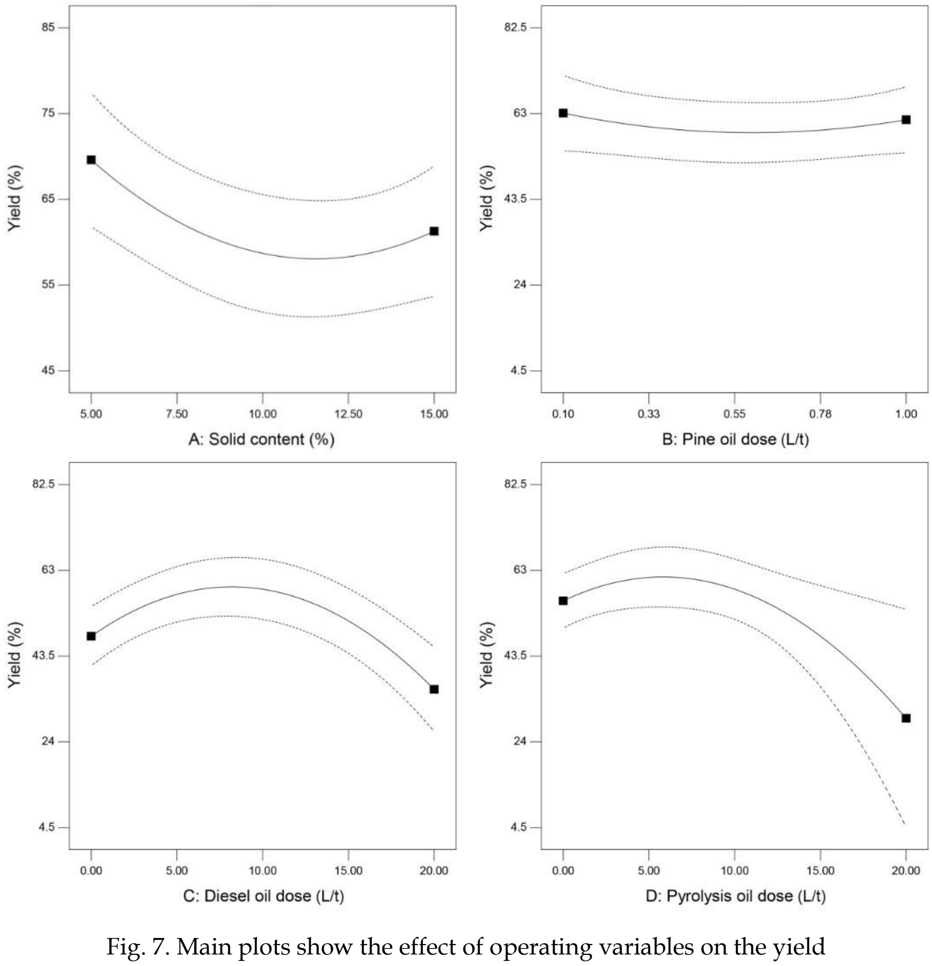


Fig. 7. Main plots show the effect of operating variables on the yield

Precious information regarding the individual effect of operating factors and their interaction can be obtained from the 3D plots of the response of a process against other independent variables (Gholami et al., 2022). Hence, experimental results were used for developing the 3D interaction plots for the flotation responses. Figures 8 and 9 depict the 3D response plots for significant interactions given in Tables 5 and 6 and indicate the nonlinear effects of all interactions on responses. Interestingly, the significant interactions are those among reagents used during the flotation experiments, i.e., pine oil, diesel oil, and pyrolysis oil. Although the individual effect of pine oil concentration on ash content was insignificant, Figure 8 clearly shows that there is a significant interaction between the pine oil and other reagents such that maximum ash content may obtain at the highest concentration of pine oil (Asgari et al., 2023). The interactions between pyrolysis oil and diesel oil for both flotation responses are very complicated. This behaviour may be attributed to two reasons; first, competitive adsorption of diesel oil and pyrolysis oil molecules may occur at the surface of particles and finally, pyrolysis oil molecules adsorption becomes dominant. Another reason may be due to possible interactions between diesel oil and pyrolysis oil molecules in the water phase such that at higher concentrations of pyrolysis oil, there would be fewer diesel oil molecules to interact with the surface of coal particles.

### 3.4. Discussion on PO-coal particles interaction

Figure 3 depicts the FT-IR spectra of coal samples before and after the addition of PO. The raw coal spectrum (Fig. 3b) exhibited a broad absorption band, indicative of the presence of hydrogen bonds in the material. The spectral regions between 1600–1300 $cm^{-1}$, 1200–1000 $cm^{-1}$, and 800–600 $cm^{-1}$ revealed the presence of hydroxyl compounds. Additionally, the absorption at wavenumbers between 3010 and 3040 $cm^{-1}$ confirmed the existence of simple unsaturated olefinic compounds. A narrow band below 3000 $cm^{-1}$ indicated the presence of aliphatic compounds, and long-chain linear aliphatic compounds were identified at 2935 and 2860 $cm^{-1}$, with supporting peaks between 1470 and 720 $cm^{-1}$ (Nandiyanto et al., 2019). Similarly, several low-intensity bands were visible between 2000 and 1700 $cm^{-1}$, repre senting simple aromatic compounds. The absorption band at 1600–1500 $cm^{-1}$ confirmed the presence of an aromatic ring with a C–H bending vibration of medium to strong intensity. In the fingerprint region (600–1500 $cm^{-1}$), a strong signal at around 1500 $cm^{-1}$ informed the aromatic ring, while a vinyl-related compound was detected at approximately 1000 $cm^{-1}$. The spectra analysis of the PO-exposed coal sample, as shown in Figure 3c revealed several key features (Mirshekari et al., 2023). The sample exhibited the presence of hydrogen bonds, which were observed along with stretching C–H signals at approximately 2925 $cm^{-1}$, as well as methine (>CH−) signals at methine C–H stretch between 2900–2880 $cm^{-1}$. Additionally, the spectra analysis also identified a C–O–H signal due to the aliphatic side groups of the amino acid residues. It's important to note that the C–O–H signal is not as strong or prominent as other signals, such as the stretching C–H or methine signals (Nandiyanto et al., 2019; Mirshekari et al., 2022).

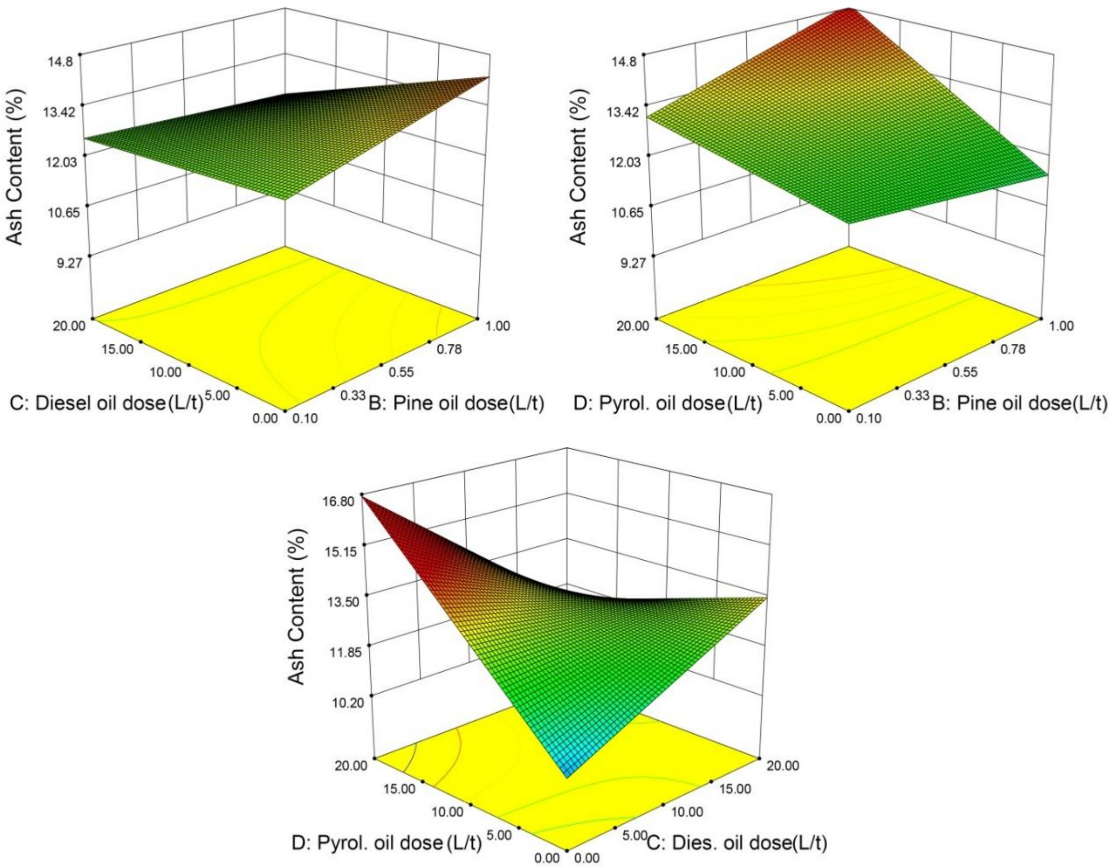


Fig. 8. 3D plots showing the interaction effects between operating variables on ash content

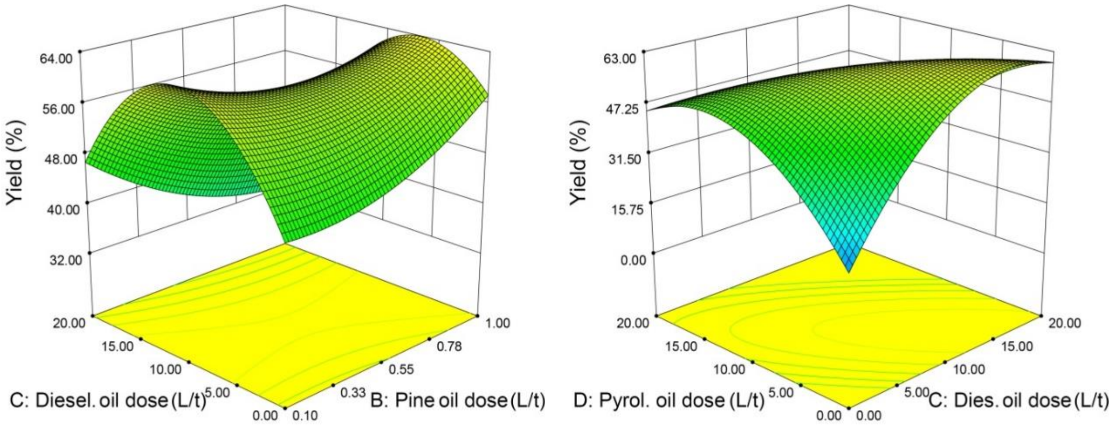


Fig. 9. 3D plots showing the interaction effects between operating variables on concentrate yield

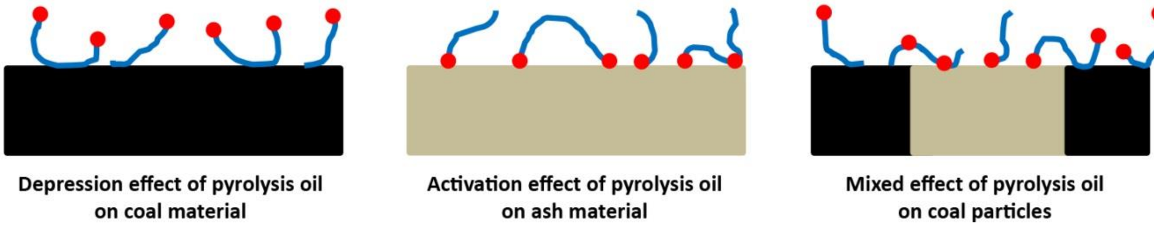


Fig. 10. Proposed mechanism for the interaction between pyrolysis oil (hydroxyl heads ●, hydrocarbon chains —) and different sites on the surface of coal particles

Generally speaking, FTIR results indicate that PO-exposed coal particles have been covered with aliphatic compounds whereas fresh coal lacks such components. Therefore, the aliphatic compounds alongside the C–H stretches increase the probability of van der Waals forces attaching the C compounds of oil to the surface carbon of the coal (Mirshekari et al., 2022). At the first look, the attachment of the PO to the coal particles seems to create a layer of oil on the surface that repels water, making the particles more hydrophobic. However, the aliphatic components may adsorb at the surface of coal particles such that the –OH groups at the end of the aliphatic chains orient toward the aquatic environment and give a hydrophilic character to the surface of coal particles. Finally, the presence of the –OH groups on the aliphatic chains in the coal particles may also increase the likelihood of attachment of other polar compounds or water molecules to the coal surface, making it more hydrophilic overall. This effect can be seen at the end of the flotation process, where more ash is produced than expected, as shown in Fig. 6, due to the increased hydrophilicity of the coal surface (Zarandi et al., 2020; Mirshekari et al., 2023). Moreover, the–OH groups in PO structure can also interact with oxide surface of as material (Table 3) so that hydrocarbon chains would then orient toward water and gave a hydrophobic character to the surface of ash material. Therefore, more ash material could be directed to froth phase by loading on rising air bubbles. The effect of PO on ash and coal materials is schematically illustrated in Fig. 10.

## 4. Conclusions

This study investigated the effect of the acetonized pyrolysis oil produced by the pyrolysis of spent-car tires as a flotation reagent in coal flotation. Individual effects and interaction of operating variables including the pulp solid content and the concentration of frother, collector and pyrolysis oil on flotation responses of ash rejection and total yield of the final concentrate were assessed. A detailed experimental design and statistical analysis were used to evaluate the significance of the impacts. Results indicated that except pine oil concentration, all other variables had a significant effect on the flotation performance. Regardless of the known effect of the pulp solid content and the concentration of diesel oil, pyrolysis oil increased the ash content in concentrate, likely due to electrostatic interaction with the hydrophilic oxide ash materials on the surface of coal particles. Additionally, by increasing the concentration of the pyrolysis oil, the total yield was decreased through a non-linear trend, it was possibly because of the competitive interaction between diesel oil and pyrolysis oil with the surface of coal particles. In spite of the unpleasant impact of pyrolysis oil on coal flotation performance, structural modifications may be useful for its potential applicability in oxidized coal flotation. Besides, more investigations are necessary to examine the clear mechanism behind the observed results of this study. In this study, the flotation behaviour of coal was assessed based on ash content and yield of concentrate that are common measures used in coal industry in Iran; however, evaluation some other criteria such as coal recovery, separation efficiency and selectivity index can also provide useful information on metallurgical effect of PO.

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