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Assessment of the Influence of Sulfuric Acid/Hydrogen Peroxide Mixture on Organic Sulfur Reduction of High Sulfur Coals and Their Chemical Composition

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Abstract

One of the contaminants in coal is sulphur. The adverse impact of sulfur on coal, such as environmental pollution, degradation of steel quality, and reduction of coal's thermal value, has led to the attention of sulfur separation methods in recent decades. Leaching (chemical dissolution) is one of the best methods for desulfurising coal, reducing sulfur in coal. In this study, hydrogen peroxide as an oxidising agent on sulfuric acid yield in reducing sulfur types of coal and chemical structure and the organic texture of high sulfur coal was investigated. The experiments were designed using a three-level response surface methodology with four duplicate points and 27 experiments. The independent variables studied were temperature, time, stirring speed and ratio of sulfuric acid to hydrogen peroxide. Dependent variables included reduction percentage of total, pyritic and organic sulfur. This study showed that 99.99% of total sulfur, 30.11% of pyritic sulfur and 69.08% of organic sulfur were reduced. These values were obtained at a temperature of 60°C, time 120 min, stirring speed 200 rpm and 3:1 ratio of sulfuric acid to hydrogen peroxide. Significant changes were observed by infrared spectroscopy (FTIR) of the coal structure before and after desulphurisation. On the other hand, the studies showed no specific changes in the bonds related to the organic coal matrix. The results showed that this method could be used as a secure process for removing inorganic and organic sulfur without destroying the organic coal matrix.

1. Introduction

As one of the most important sources of energy worldwide due to its exploitation and utilisation, coal causes increasingly severe ecological problems, especially air pollution.

One of the significant causes of pollutant emissions is the sulfur compounds produced by coal combustion. Additionally, if coke is made from coal, some sulphur is retained in the coke, which precipitates on the surface of the iron crystals during the steelmaking process, resulting in the fragility of the generated steel and adverse impacts on its qualities [1] [2] [3] [4].

The sulfur contents of coal vary from 0.3% to 0.5% on average. Consequently, sulfur contents are more variable and tend to be higher. Sulfur is often present in coal in both organic and inorganic forms, depending on coal [5] [6]. Inorganic sulphur is often found in two forms: disulphide and sulphate [7] [8]. The organic form of sulphur, directly attached to the organic coal matrix, is typically found as thiols, sulphides, disulfides, thiophenes, cyclic sulphides, and organic sulphur removal is one of the most difficult challenges in coal desulphurisation [9] [10].

The sulfur dioxide in the air combines with rainwater to form sulfurous acid. Acid rain resulting from sulfur dioxide harms the environment and destroys the ecological balance. Coal, before utilisation, is exposed to three ways: physical, chemical, and microbial, or a combination of these techniques [11] [12] [13], Chemical techniques, such as reacting coal with carbon disulfide, are used to remove the organic and some of the inorganic sulfur. As reported, chemical-related desulphurisation of coal with various oxidising agents, acid/alkaline leaching, electrochemical reduction, solvent extraction, pyrolysis, air oxidation, and microwave/ultrasonic are adequate for the removal of ash and pyretic and sulfate sulfur but yield only a slight reduction of organic sulfur [14] [15] [16].

Inayat *et al.* investigated the reduction and control of sulfur using coal pretreatment methods such as the oxy-desulfurisation technique [17] [18] [19]. The coal samples utilised in their research were obtained from the Prince of Wales mine in Yorkshire (United Kingdom); the influence of air and oxygen pressure on sulphur reduction was investigated by maintaining a constant time constant across a varied amount of time in this study [3] [16]. Sulfur reduction is prevented by increasing the quantity of oxygen in the air, and carbon reduction is prevented by raising the amount of oxygen in the air 100 bars of pressure [20] [21]. The desulphurisation of selectively oxidised coals and unoxidised coals were investigated using pyrolysis and various base agents [22] [23]. The coal samples utilised in their research were from the Illinois Basin Coal Sample Pro-

gram, established in 1989. The independent variables studied were temperature, time, stirrings, and oxidation level pyrolysis pressure during the preparation stage [24] [25].

In some cases, sulfur removals of up to 95% were obtained [26] [27]. Most of the inorganic sulfur and small organic sulfur content was reduced, and the ash was significantly lowered. The coal samples utilised in their investigation came from Thailand, where they were mined [28] [29] [30]. The process was performed under certain conditions (temperature, time, and a particular concentration of hydrogen peroxide and sulfuric acid) [31] [32]. [33] studied the desulphurisation of high-sulfur coals using various inorganic reagents. The coal samples utilised in their study were from India, and they were analysed by the researchers. The results showed that hydrogen peroxide, the most suitable agent with 50 wt% - 90 wt%, was used to reduce sulfur under specific temperature conditions with a time of 10 - 30 min. Organic sulfur has been severely reduced, but the proportion of sulfate and pyrite sulfur has decreased significantly compared to organic sulfur [9] [14]. Mohammad Reza Ehsani examined a coal sample with a high caking index in the Tabas region that contained a small amount of ash. In this study, Tabas coal desulphurisation was performed in a laboratory reactor with different reagents such as $Fe_2(SO_4)_3$, $FeCl_3$, $NaOH$, CH_3OH, HNO_3, and H_2O_2 under pressure and temperature [20] [34]. The results showed that ferric sulfate is the most suitable chemical reagent for the desulphurisation of coal in the Tabas region, which can reduce the components of pyrite sulfur. Hydrogen peroxide with concentrations of 10% and 20% was also used for 1 hour, which reduced total sulfur and ash to 30.96%, and 45.24%, respectively [35] [36]. Nam *et al.* performed a thermal-treatment process (pyrolysis and oxidation) at a temperature of 550°C and a reaction time of 15 - 20 min, and optimal conditions for the process of leaching with 30% hydrogen peroxide at 90°C for 60min. These processes show that in these conditions, the highest reduction in total sulfur and organic sulfur was 54% and 15%, respectively, and the coal samples used in this study were collected from Australia and South Africa [8]. Ahmed *et al.* obtained a sample of Pakistan's Lakhara mine, which leached with hydrogen peroxide/sulfuric acid under conditions (time, temperature, and particle size) that pyrite sulfur decreased by 25% [37] [38]. Samit Mukherjee *et al.* investigated the effect of hydrogen peroxide solution alone and in the presence of dilute sulfuric acid for the desulphurisation of coal collected in the Makum coalfield situated at the North-Eastern region, India [39]. The results showed that Hydrogen peroxide (15) alone leads to removal of over 76% pyritic sulfur and around 5% organic sulfur at 25°C, which increases to the almost complete removal of pyritic and sulfate sulfur and over 26% organic sulfur in the presence of 0.1 N H_2SO_4 [24] [40]. The presence of organic sulfur in coal in the Tabas region makes it difficult to use on an industrial scale. Because of this, a chemical approach was used in this research in an effort to reduce the amount of sulphur in coal. The influence of several factors such as temperature, time, mixer speed, and

concentration of sulfuric acid and hydrogen peroxide on the quantity of desulphurisation was also investigated [41] [42] [43]. Assessment of the influence of Sulfuric Acid/Hydrogen Peroxide mixture on organic sulfur reduction of high sulfur coals and their chemical composition is the main purpose of this research [44].

2. Experimental Procedure

2.1. Materials

The coal samples for this research were collected from the Tabas coal mine, located in. (I don't know where this mine is); and all samples were examined under ISO334 and ISO157 tests to obtain the amount of sulfur, Equipment used includes: Polarizer (Ipek.4500-USA), UV-Vis ultraviolet spectrometer (Ipek.4500-USA), Shaking Screen For Recycle Sandblasting Media D1200Mm-1S-C-USA, Fourier-transform infrared spectroscopy (Perkin Elmer BX Spectrometer-USA) conversion. In this study, sulfuric acid 98%, hydrogen peroxide 30%, hydrochloric acid 37%, nitric acid 65%, ammonia 25%, standard iron, magnesium oxide, sodium carbonate, methyl orange, barium chloride, potassium sulfate, hydroxylammonium chloride and, sodium acetate which are high purity compounds (99%) and are manufactured by Merck, have been used to reduce sulfur in the desulphurisation process. Also, Congo red and phenanthroline have been used as an indicator to determine sulfur by ISO standard method [45] [46] [47]. The ultimate and proximate analyses of the coal sample used are summarised in Table 1. The amounts of the organic sulphur present are also indicated.

2.2. Sample Preparation

Coal samples were crushed and ground to below −75μm particles, which were dried at the temperature 105°C ± 5°C in an electric oven, and then it was collected and sealed for the subsequent experimental investigations. The analyses of the coal samples are presented in Table 1. The ash, moisture, and volatile matter percentages were estimated using ISO1171, ISO597, and ISO562 methods? For determining all sulfur forms, this international standard has been used. The ISO334 is used for total sulfur (Eschka Method), and ISO157 is used for sulfate and pyritic sulfur determination [46] [47] [48].

Recordings were made using the KBr pellet method FTIR spectra in the range 4000 - 400 cm^{-1} with a spectral resolution of 2 cm^{-1} in the absorption mode using an F.T./IR-Perkin Elmer BX Spectrometer in the range 4000 - 400 cm^{-1} with a spectral resolution of 2 cm^{-1} in the absorption mode. Pelleted samples were

Table 1. Characteristics of the coal samples (wt%).

Coal	Sulfur components				Proximate analysis (dry basis)		
	Sulfate	Organic	Pyrite	Total	V_m	Ash	M
	0	2.7	1.2	3.9	69.88	20.05	0.54

made by combining 2 mg of the powdered sample with 200 mg potassium bromide (KBr) [16] [49] [50]. The procedures are illustrated in the flow chart shown in **Figure 1**.

3. Results and Discussion

The investigation of the industrial application of coal is the most critical aspect of using this valuable mineral, so in order to optimise the experiments and analyse the accuracy of the results, the Expert Design software versions 7 and 11 were utilised using the response surface method, D-optimal of section and four temperature factors from 20°C to 60°C, time from 30 to 120 min, mixer speed from 200 to 500 revolutions per minute and the ratio of sulfuric acid to hydrogen peroxide 1:1, 3:1 and 3:2, number 27 experiments were performed. The basis for selecting these parameters is the optimisation of the experiments and placing in the range that gives the most reduction of organic sulfur, this is done according to past research, and the reason for using the response surface method is that using this method can be at least the number of experiments yielded the best and most practical results with the least amount of consumables. The present study results on Tabas coal samples, with an initial sulfur content of 3.9%, are presented in **Table 1**. Sulfuric acid and hydrogen peroxide were present at 20 percent and 15 percent concentrations, respectively [51] [52] [53]. The percentage reduction sulfur forms in coal samples after the desulphurisation process with various reagents are presented in **Table 2**.

All of **Figures 2-8** are drawn based on the results presented in the above table to determine the better effect of the parameters. The effect of temperature on the reduction of organic sulfur is shown in **Figure 2**.
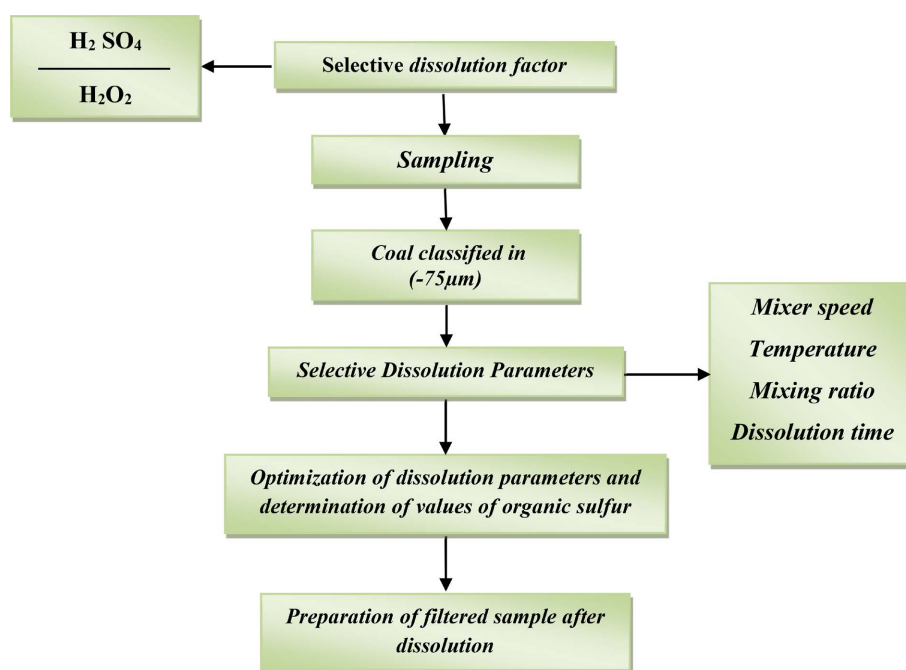

Figure 1. Flowchart of desulfurization process.

Table 2. Percentage reduction of sulfur after desulphurization.

No. Sample	S_t	S_p	S_o	Recovery
1	90.43	47.37	43.06	93.14
2	99.33	46.03	53.3	91.10
3	92.92	37.87	55.05	90.47
4	93.79	56.18	37.61	94.37
5	98.14	59.42	38.72	89.93
6	99.34	38.47	60.87	87.64
7	87.06	40.68	46.38	88.90
8	87.36	31.28	56.08	91.87
9	76.25	20	56.25	87.37
10	55.27	30.77	24.5	90.45
11	99.02	52.06	46.96	85.12
12	73.46	38.33	35.13	92.43
13	88.68	25.93	62.75	91.46
14	75.44	38.26	37.18	92.52
15	84.11	33.81	50.3	86.74
16	99.37	59.37	40	89.99
17	56.75	28	28.75	93.12
18	90.46	42.11	48.35	86.23
19	98.34	31.54	66.8	92.43
20	76.65	33.98	42.67	91.65
21	71.67	38.3	33.37	92.47
22	60.82	29.77	31.05	91.41
23	37.18	19.12	18.06	92.39
24	69.08	35.53	33.55	85.88
25	97.57	34.11	63.46	86.86
26	99.99	30.91	69.08	86.25
27	92.04	55.02	37.02	95.96

It is observed that the amount of organic sulfur decreases with increasing temperature. According to **Figure 3**, with increasing temperature, a decrease in organic sulfur is observed from 14.45% to 55.27%. This factor has also been influential in the interaction with the time and ratio of sulfuric acid to hydrogen peroxide.

As shown in **Figure 4**, the rate of decrease in organic sulfur has increased with enhancing the time of dissolution, and this factor has been influential in the interaction with temperature.
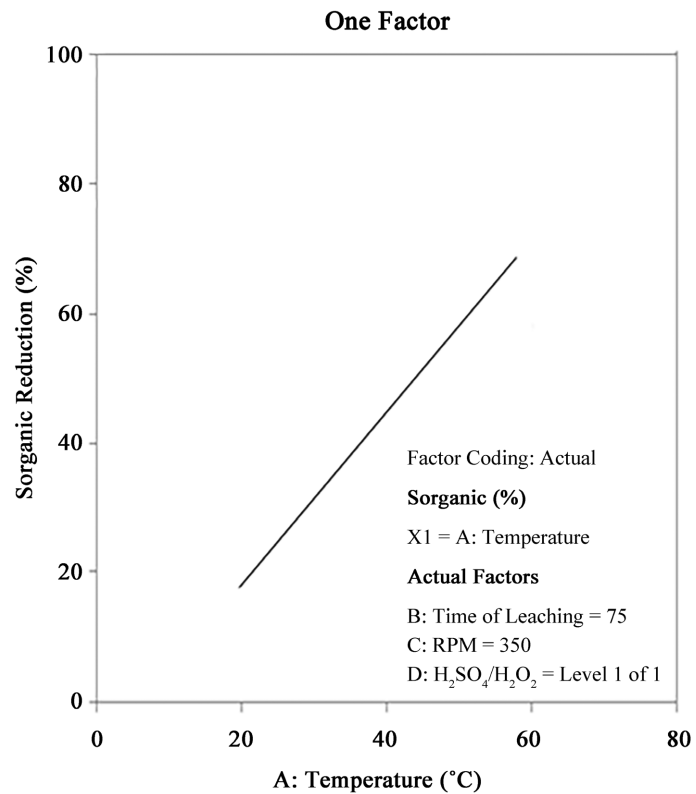
Figure 2. Effect of temperature on reduction organic sulfur (Time = 75 min, Mixer speed = 350 revolutions per min and the ratio of sulfuric acid to hydrogen peroxide = 1:1).
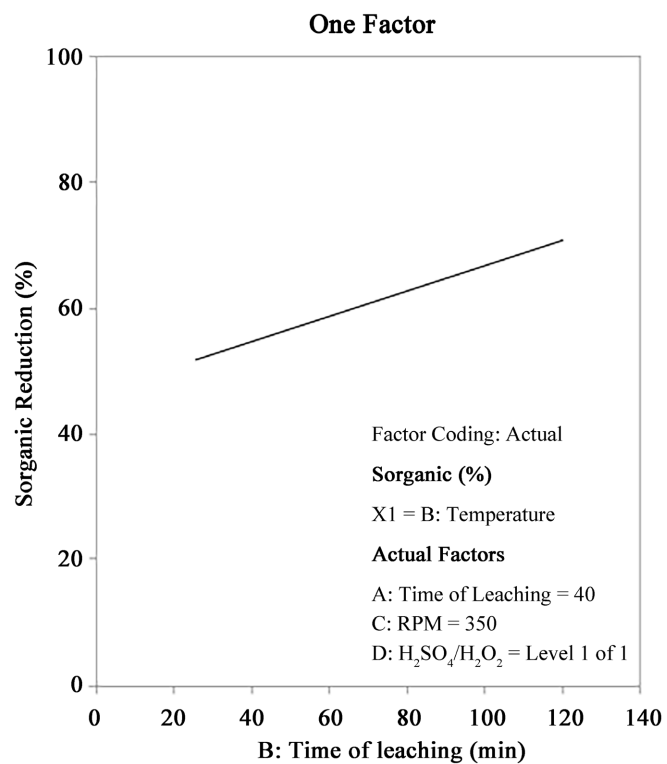

Figure 3. Effect of time on reduction organic sulfur (Temperature = 40°C, Mixer speed = 350 revolutions per min and the ratio of sulfuric acid to hydrogen peroxide = 1:1).

As shown in **Figure 4**, increasing the amount of organic sulfur has not changed with increasing the stirrer speed, and it almost follows a constant trend, and this factor has also been influential in the interaction with the ratio of sulfuric acid to hydrogen peroxide.

As shown in **Figure 5**, as the ratio of sulfuric acid to hydrogen peroxide increases, the rate of decrease in organic sulfur increases first and then decreases.

So that the most significant effect on desulphurisation is related to the ratio of 3:1, and the ratios of 3:2 and 1:1 are in the following positions. This factor has been influential in the interaction with temperature and mixer speed.

Sulfuric acid to water (green and red curves) rises with decreasing temperature, as seen in the above image when the ratios of 3:1 and 1:1 are used. Organic sulphur decreases when the ratios are used. However, in the 3:2 ratio of sulfuric acid to hydrogen peroxide (blue curve) with increasing temperature, a significant decrease in organic sulfur reduction is seen, and it is because the ratio of hydrogen peroxide has increased. It can be said that the oxidation agent is likely increased, and due to surface oxidation, part of the organic sulfur has lost the available surface for the leaching agent. Therefore unwanted oxidation is recommended as an annoying factor for reducing organic sulfur in this situation (Proportion 3:2) that we have a passive layer here (**Figure 6**).

The rate of decrease in organic sulfur has not changed much with increasing time of dissolution in **Figure 7**. According to the figure, with the increase in
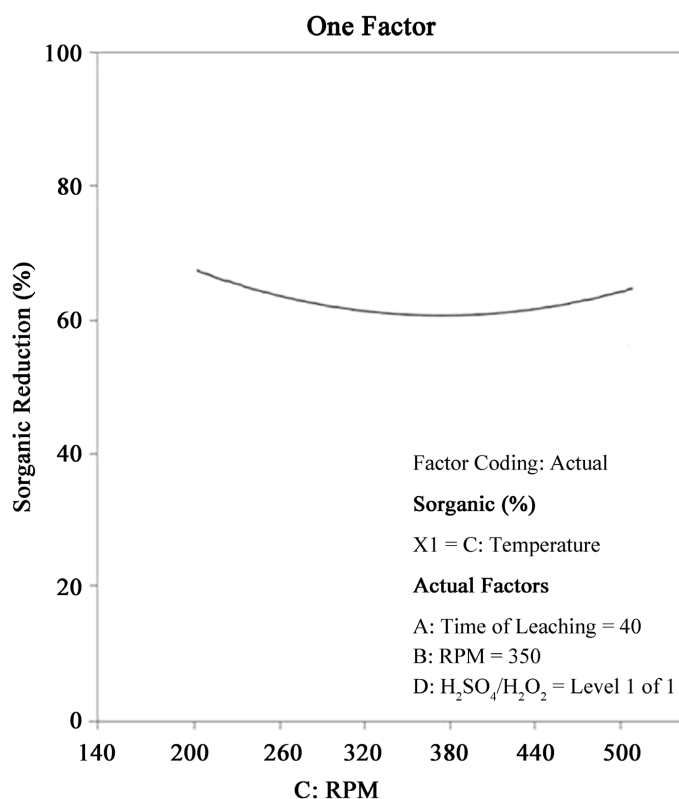

Figure 4. Effect of mixer speed on reduction organic sulfur (Temperature = 40°C, time = 75 min and the ratio of sulfuric acid to hydrogen peroxide = 1:1).
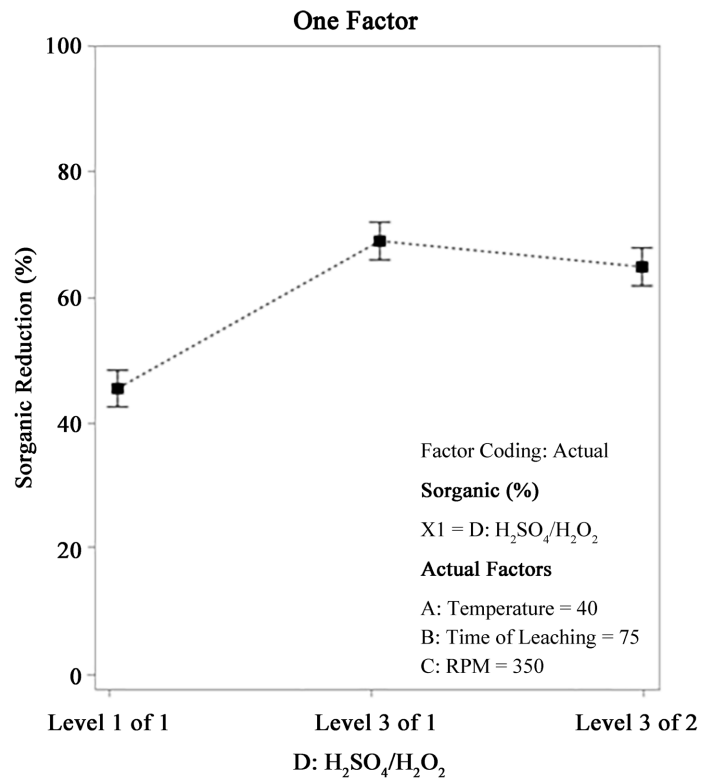
Figure 5. Effect of ratio of sulfuric acid to hydrogen peroxide on reduction organic sulfur (Temperature = 40˚C, time = 75 min and mixer speed = 350 revolutions per min).
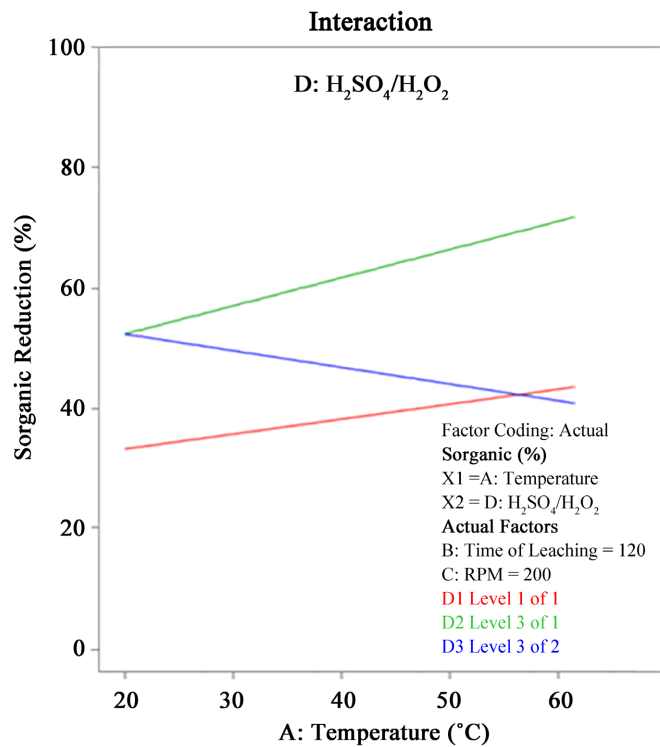

Figure 6. Effect of temperature interaction ratio of sulfuric acid (green) to hydrogen peroxide (blue) on reduction organic sulfur (water is red) (Time = 120 min, Mixer speed = 200 revolutions per min and the ratio of sulfuric acid to hydrogen peroxide = 1:1, 3:1 and 3:2).
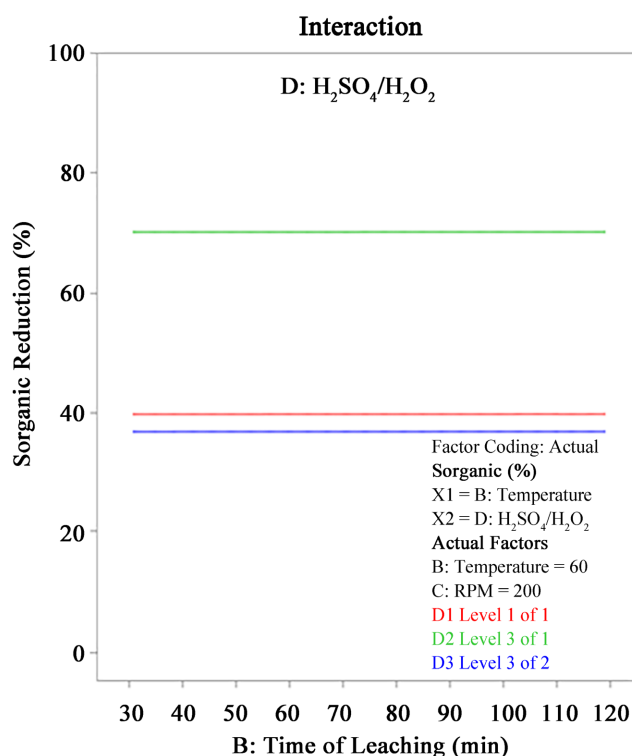
Figure 7. Effect interaction of time ratio of sulfuric acid to hydrogen peroxide on reduction organic sulfur (Time = 75 min, Mixer speed = 200 revolutions per min and the ratio of sulfuric acid to hydrogen peroxide = 1:1, 3:1 and 3:2).

time from 30 to 120 min, the reduction rate of organic sulfur did not change, but the ratio of 3:1 compared to 1:1 and 3:2 has decreased. The more significant effect of the 1:1 ratio indicates that if we want to do more research between the 1:1 and 3:2 ratios, it is essential to note that with a slight difference in time interaction over this parameter, the situation improved by 1:1. However, it is not very important, and the difference is slight due to the significant difference in the ratio of 3:2 to 1:1 (**Figure 7**).

As shown in **Figure 8**, with increasing mixer speed, the rate of decrease in organic sulfur for the 3:1 and 1:1 ratios was almost constant and increased for the 3:2 ratio. This parameter reduces the oxidation properties of the surface and eliminates the passive layer created in the ratio of 3:2. Removal of the passive layer created in the 3:2 ratio.

FTIR analysis was used to determine the functional groups of coal samples, and the results were compared with the standard coal samples. The basis for selecting samples was comparing the amount of organic sulfur (maximum, which is S26, the minimum, which is S23 and average, which is S15) and examining the amount of reduction calculated and the performance of the solvent agent in the samples. The result of this analysis is in the form of the spectrum shown in **Figure 9**. Several spectral regions show the quality of the leaching coal sample and the raw coal sample and their functional groups.

The 3300 to 3700 cm^{-1} is the widest band in all coal samples. These bands are
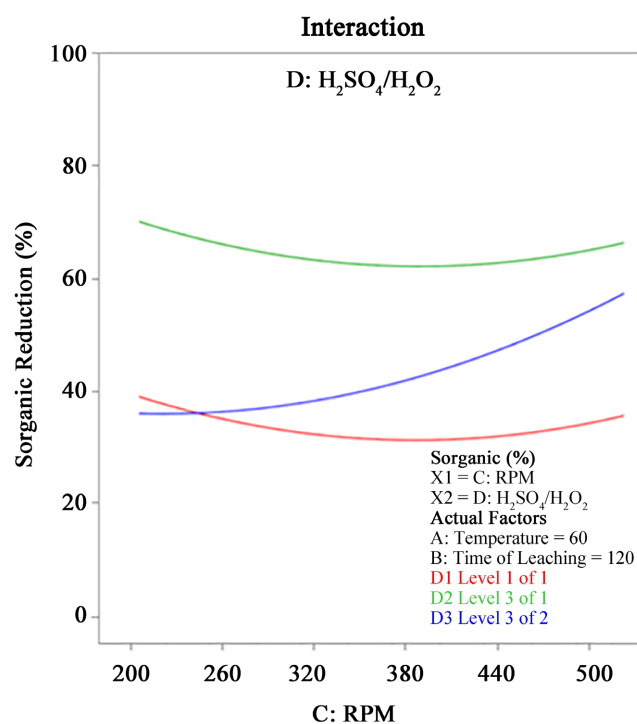
Figure 8. Effect interaction of mixer speed and ratio of sulfuric acid to hydrogen peroxide on reduction organic sulfur (Temperature = 60˚C, time = 120 min, and the ratio of sulfuric acid to hydrogen peroxide = 1:1, 3:1 and 3:2).
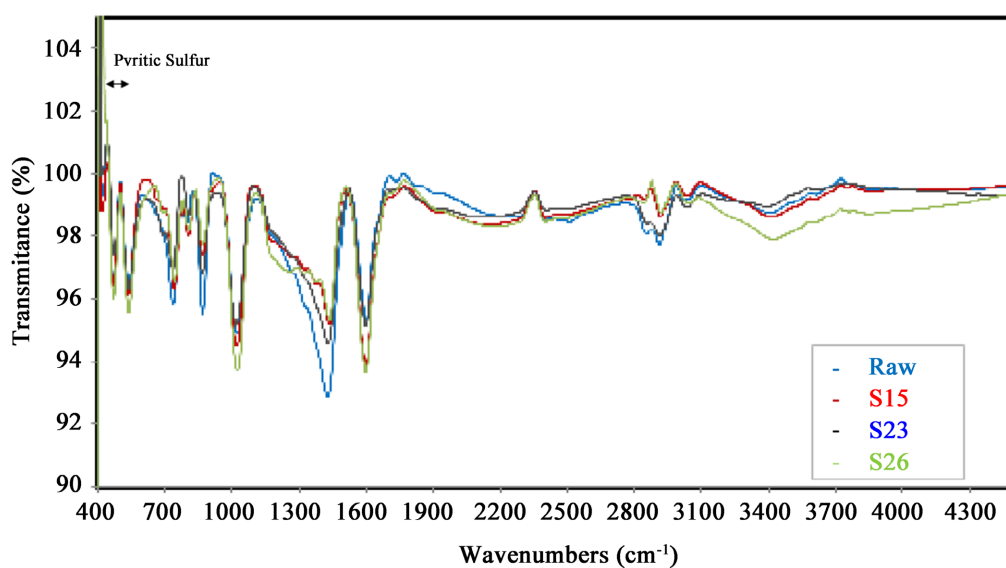

Figure 9. FTIR spectroscopy on raw samples and leached samples.

attributed to the organic stretching of O-H groups. Peaks wider than the O-H stretching vibration may be associated with high ratios of O/C and H/C.

In the raw and leached specimens, the band of absorption range of 2850 to 2930 cm^{-1} is attributed to the symmetrical and asymmetrical stretching vibration of C-H methyl and methylene from cyclic hydrocarbons of non-tensile aliphatic.

Besides the disappearance of the peaks of minerals, several new organic peaks

appear for processed coal, including peaks of aromatic C=C stretching and -C.O.O. aromatic asymmetric of stretching groups of at 1400 to 1600 cm^{-1} of the samples, respectively.

Organic sulfur peaks are not visible in the spectrum, while mineral groups (1000 to 1200 cm^{-1} and 400 to 600 cm^{-1}) are present in compressed coal. Sulfur-containing minerals show a band of about 430 to 500 cm^{-1}, attributed to pyrite.

As can be seen in the spectrum, decreasing sulfur has caused significant changes in peaks related to pyrite, other minerals and organic matrix bonds. As a result, this method can be used to reduce sulfur without destroying the organic carbon matrix.

4. Conclusions

Examining the results of experiments and determining the influential factors on reduction sulfur forms of coal, it was found that the combination of sulfuric acid in the presence of hydrogen peroxide as a leaching agent to reduce coal sulfur is effective and by this acidic factor can be the amount significantly of sulfur reduced depending on the type of sulfur and provided a possibility of reducing 99.99% of total sulfur, 30.11% of pyrite sulfur and 69.08% of organic sulfur.

By optimising the results, the best solution for the maximum of total sulfur reduction is temperature 60˚C, time 120 min, mixer speed 200 rpm and the ratio of sulfuric acid to hydrogen peroxide 3:1, which is a reduction of 96.82% of total sulfur, 29.41% pyrite sulfur and 67.41% organic sulfur are expected.

By optimising the results, the best solution for the maximum of pyrite sulfur reduction is temperature 60˚C, time 120 min, mixer speed 200 rpm and the ratio of sulfuric acid to hydrogen peroxide 3:1, which is the reduction of 97.33% of total sulfur, 28.08% pyrite sulfur and 69.25% organic sulfur are expected.

By optimising the results, the best solution for the maximum of organic sulfur reduction is temperature 60˚C, time 120 min, mixer speed 200 rpm and the ratio of sulfuric acid to hydrogen peroxide 3:1, which is the reduction of 90.84% of total sulfur, 30.51% pyrite sulfur and 60.33% organic sulfur are expected.

Organic sulfur peaks are not visible in the spectrum, while sulfur-containing minerals show a band of about 430 to 500 cm^{-1}, attributed to pyrite. The sulfur decreasing process has caused significant changes in peaks related to pyrite, other minerals and organic matrix bonds.

Due to the lower price of sulfuric acid and hydrogen peroxide as a leaching agent and the possibility of performing this process at low temperature and low time, it has made sulfuric acid more economical in the presence of hydrogen peroxide. As a result, it can be used in industry as a method with good efficiency with high ability to reduce organic sulfur.

Acknowledgements

Conflicts of Interest

The authors declare no conflicts of interest regarding the publication of this paper.

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