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Desulfurization of Tabas coal with microwave irradiation/peroxyacetic acid washing at 25, 55 and 85 °C

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Abstract

Tabas coal sample from Iran was desulfurized with combination of microwave irradiation and peroxyacetic acid washing in a batch reactor. The effect of irradiation time and power, peroxyacetic acid reaction time and temperature and particle size on reduction of sulfur was investigated. For microwaved/acid washed samples, depending on desulfurization conditions, the reduction in pyritic, organic and total sulfur ranged from 26 to 91, 2.6 to 38.4 and 17 to 65%, respectively. Microwave irradiation had positive effect on desulfurization with peroxyacetic acid. For < 300 μm size fraction, after irradiation, the reduction enhanced from 49.9 to 86.6% in pyritic sulfur, 23.8 to 35% in organic sulfur and 36 to 61.9% in total sulfur. FT-IR spectroscopy of coal structure before and after desulfurization has shown significant changes in the peak corresponding to pyrite whiles the bands related to coal organic matrix have not changed significantly. The results prove that this method can use as secure process on removal of inorganic and organic sulfur without destruction of coal organic matrix.

Keywords: Coal desulfurization; Inorganic sulfur; Organic sulfur; Microwave irradiation; Peroxyacetic acid

1. Introduction

Coal is an economical source of energy in the world. In recent decades one of disadvantages associated to use of coal as energy on combustion systems is emission of sulfur oxides and hydrogen sulfide in environment. In steel making industry coking process cannot remove sulfur from coal and it combines with molten iron in blast furnace. The sulfur sediments on the iron crystal surface make steel more brittle and decrease its plasticity property. These problems have led to use of fluidized bed combustion and flue gas desulfurization process in industry to control sulfur emission. However, problems with the existing technology concerning costs, efficiency, applicability and waste disposal have led to increase research on pre-combustion desulfurization process which have the potential of solving of this problems [1].

Sulfur in coal occurs in the forms of inorganic and organic. The inorganic sulfur is present mainly in two forms, as disulfides (pyrite and marcasite) and sulfate (mainly calcium, iron and barium). The organic form, which is bound directly to the organic coal matrix, generally occurs in forms of thiols, sulfides, disulfides, thiophenes and cyclic sulfides [2]. Recently the presence of secondary sulfur in coal has been reported that in which the iron have bonded with organic sulfurs in aromatic and aliphatic systems [3].

Microwaves are electromagnetic waves with frequency range of 300 MHz–300 GHz, between infrared and radio frequencies [4]. Microwave ovens generally operate at a frequency of 2.45 GHz corresponding to a wavelength of 12.2 cm and energy of 1.02×10^{-5} eV for domestic and industrial applications [5]. Materials are different in absorbing microwave radiation; some of them absorb this waves, are called dielectrics, whilst others appear transparent to or reflect microwaves [6] (Fig. 1). Previous studies concerning dielectric properties of organic and inorganic material in coal show differences in dielectric characteristics and accordingly differential heating of mineral and organic material [7–9].

Chatterjee et al. [10] have shown the dielectric constant for dry coal, pyrite and mineral matter (without pyrite)

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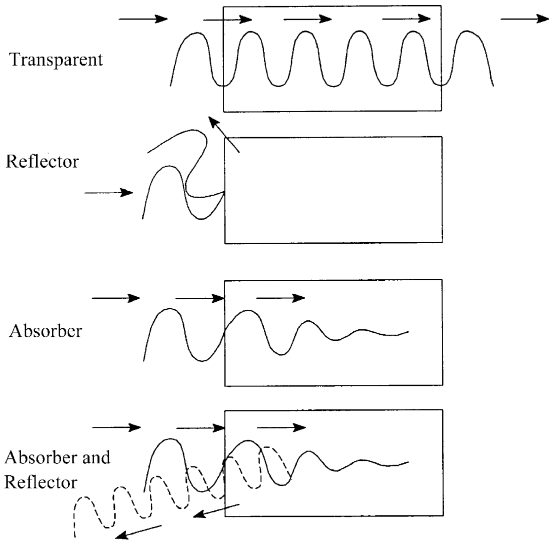

Fig. 1. Material responds to microwave irradiation [6].

are 3, 7 and 4.6, respectively. Weng [11] has shown that by exposing coal to microwave irradiation, localized heating occurs at pyrite sources and pyrite decompose according to the following reaction

$$FeS_2 \rightarrow Fe_{1-x}S \rightarrow FeS \text{ or } FeSO_4 \qquad (0 < x < 0.125) \quad (1)$$

The reaction was found to progress from left to right as irradiation time increased. In this process the inorganic sulfur removal was from 5 to 44% for irradiation times of 30–100 s. With combination of the process with HCl (5%) acid washing, the acid has attacked the FeS to form H_2S and a 97% decrease in inorganic sulfur was obtained [11].

Also according to the process described by Zavitsanos and Bleiler [12] in response to the effect of the microwave irradiation the bonds of sulfur–iron in pyrite and sulfur–carbon in organic sulfurs are broken and some of sulfur is released and changed to gaseous, reacted elements through molecular bonding.

Use of peroxyacetic acid as an oxidative reagent for desulfurization of coal have reported by Palmer et al., Sonmez et al. and Aelst et al. [13–17]. This reagent is believed to produce the hydroxyl cations that are strong electrophiles and react with sulfur atoms because they are considerably more nucleophilic than carbon atoms. It was found that this process is able to remove inorganic sulfur, however, only 10–25% of the organic sulfur could be removed without high dissolution of coal.

Since the C–S and Fe–S bonds of inorganic and organic sulfurs in microwaved coal is weaker than of unmicrowaved, it seems these coals would be easier to desulfurize after irradiation pretreatment.

In this paper microwave irradiation has used not only for desulfurization process but also as a pretreatment for subsequent desulfurization with peroxyacetic acid, then this work is different from previous published work. The objective of this work is to study the effect of power and time of irradiation for microwave process and temperature, reaction time and coal particle size for peroxyacetic acid extraction, on the extent of removal of different forms of sulfur from Tabas coal.

2. Experimental

2.1. Coal sample characterization

The bulk sample (600 kg) was collected from all active stopes in C1 seam of Tabas coal mine in Iran. The sampling techniques similar to those of Jones riffles and conning and quartering methods were adopted and representative samples were prepared for further studies. Proximate and ultimate analysis of representative sample has been carried out according to standard methods. Total, pyritic and sulfate forms of sulfur in all samples have been determined by ISO methods 334 and 157 in duplication [18,19]. For pyritic, the iron concentrate in nitric acid solution has been determined by colorimetry alternative. Organic sulfur was determined by difference. The XRD studies have been carried out to determine the mineralogical composition of sample. The results are shown in Table 1.

The optical microscopic investigation shows that pyrite is present in the forms of discrete grains, fracture and cavity filling, regular and irregular framboid with average particle size of 1–30 μm. The point analysis of iron and sulfur that prepared with SEM to determine the distribution of sulfur on coal maceral and lithotypes are shown in Fig. 2. According to the dotted Fe and S, pyrite is distributed even finer than 1 μm. Removal of this fine distributed sulfur from coal needs to fine grinding and is impossible with conventional methods in practice.

Table 1
Characterization of Tabas coal representative sample

Proximate analysis (wt% as received)	
Moisture	0.75
Ash	32.3
Volatile matter	20.12
Fixed carbon	46.83
Ultimate analysis (wt% daf)	
C	86.25
H	4.31
N	2.45
S	0.67
O_{diff}	6.32
Forms of sulfur (wt% db)	
Total	1.44
Pyritic	0.77
Sulfate	0.0
Organic	0.67
Mineralogical composition	
Illite, Quartz, Kaolinite, Goethite, Feldspar, Calcite, Pyrite, Hematite	

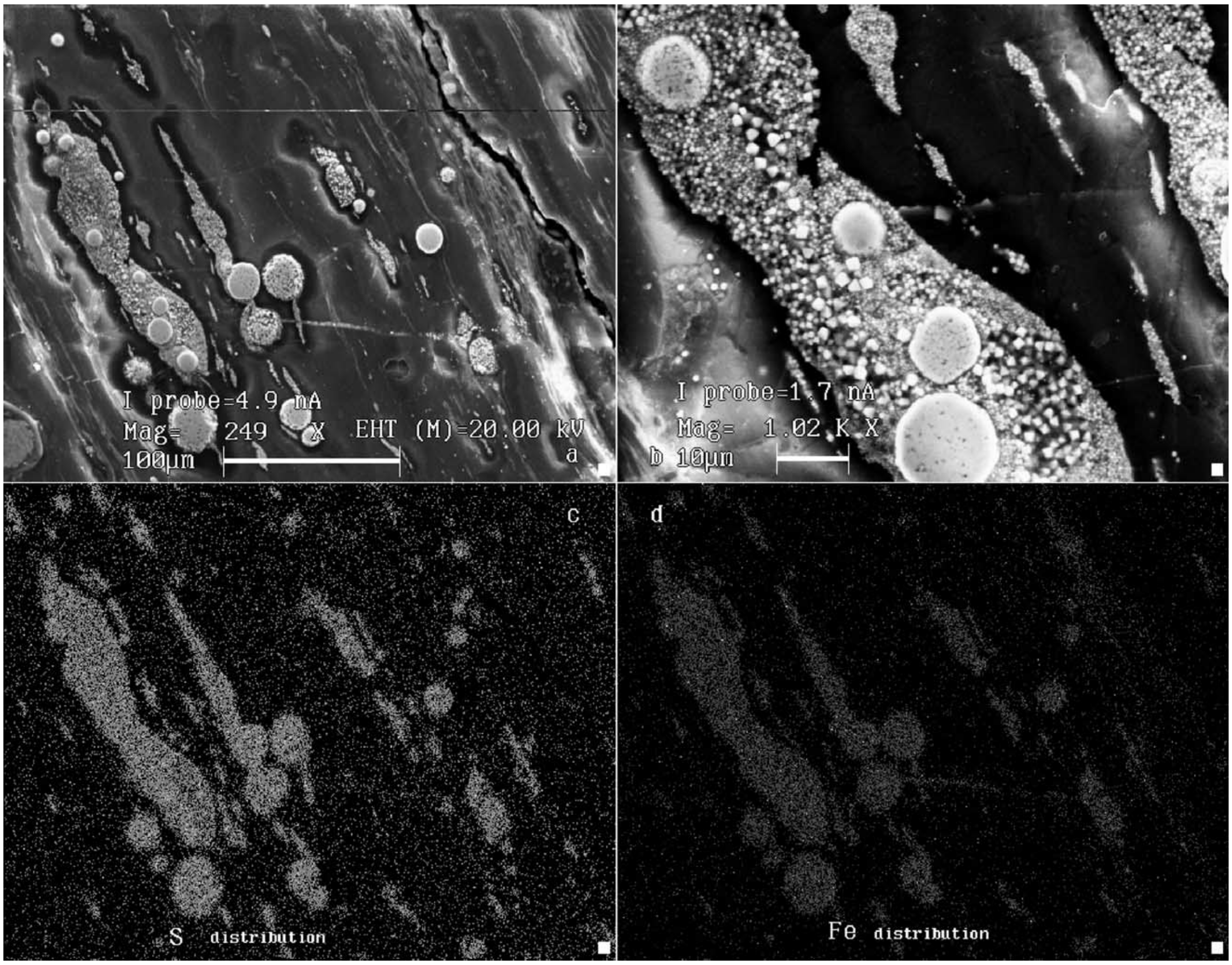

Fig. 2. Microphotographs, SEM: (a) and (b) framboidal distribution of pyrite; (c) S distribution; (d) Fe distribution.

2.2. Microwave irradiation

In microwave experiments, about 30 g coal was ground to below 1400 μm and has been spread with thickness of 0.5 cm in a Pyrex container. Microwave irradiation was carried out in a Butane microwave oven with 2.45 GHz frequency and 0–1000 W energy. The dimension of oven was $556 \times 313 \times 429$ mm^3 with internal capacity of 32 l and equipped to air circulation system. The irradiation times of 50, 80 and 110 s have been used in different powers at 600, 800 and 1000 W. The microwaved samples cooled in desiccator and were weighted and analyzed for total, sulfate, pyritic and organic sulfur. Percentage changes in sulfur in comparison to the original values were calculated as described in literature [20].

2.3. Peroxyacetic acid washing

After microwave irradiation, the sample on optimized operating conditions (time and power) was used to further desulfurization with peroxyacetic acid. The procedure involved dispersing 8 g of coal in 240 ml of glacial acetic acid and warming it to desired temperature and then adding 80 ml of H_2O_2 solution (30% w/v) [15]. The experiments were done in a 750 ml Pyrex reactor equipped with a thermometric tube and stirring. The reaction times of 30, 60, 90 and 120 min, particle sizes of 850–1400, 300–850 and <300 μm and temperatures of 25, 55 and 85 °C were used in the process. After reaction the reactor was cooled and filtered to recover of leached coal. The filtrate washed with hot water and dried in oven at 100 °C for 3 h and analyzed for pyritic and total sulfur. All of chemical reagents were obtained from Merck manufacture.

3. Results and discussion

3.1. Effect of irradiation time and power

The results of microwave irradiation on sulfur reduction are presented in Table 2. Initially, sulfur reduction was low but with increasing of power and time to 1000 W and 80 s sulfur removal increased to about 19%. Therefore the extended irradiation time and power is necessary for

Table 2
Effects of microwave power and time on desulfurization of Tabas coal (− 1400 μm)

Power (W)	Time (s)	S content (db%)				S reduction (%)			Coal yield (%)
		Sulfate	Pyritic	Organic	Total	Pyritic	Organic	Total	
Unmicrowaved coal	–	0	0.77	0.67	1.44	–	–	–	–
600	50	0.063	0.72	0.647	1.43	7.17	4.13	1.42	99.27
	80	0.054	0.69	0.606	1.35	11.16	10.33	7.05	99.14
	110	0.053	0.66	0.627	1.34	15.06	7.27	7.79	99.09
800	50	0.035	0.67	0.635	1.34	13.73	6.04	7.74	99.14
	80	0.038	0.6	0.642	1.28	22.82	5.1	11.96	99.04
	110	0.026	0.57	0.624	1.22	26.77	7.87	16.19	98.92
1000	50	0.027	0.61	0.643	1.28	21.53	4.94	11.95	99.05
	80	0.032	0.53	0.618	1.18	31.89	8.73	18.91	98.95
	110	0.027	0.52	0.653	1.2	33.21	3.62	17.59	98.89

enhancing the level of desulfurization. Some of pyritic and organic sulfur has transformed to sulfate form. It can be seen the removal of inorganic sulfur has increased with enhancing power and time. It is subsequence of localized heating on pyrite particles that decompose its structure and convert it to FeS, $FeSO_4$ and gaseous compounds. Also the removal of organic sulfur varied from 3.62 to 10.33%. It suggests with increasing of power and time some of organic sulfur converted to other forms and do not remove from coal organic matrix.

In this process the reaction between organic and inorganic sulfur and coal organic matrix take place as follows [12]

$$S(bound) + H_2O(absorbed) + \Delta H \rightarrow H_2S(gas) \quad (2)$$

$$S(bound) + H(bound) + \Delta H \rightarrow H_2S(gas) \quad (3)$$

$$S(bound) + O(bound) + \Delta H \rightarrow SO_2(gas) \quad (4)$$

$$S(bound) + CO_2(bound\ or\ gas) + \Delta H \rightarrow COS(gas) \quad (5)$$

Sulfur to be separated upon the forming of hydrogen sulfide, sulfur dioxide or sulfur carbonyl. Bound in these reactions means sulfur bound to iron (as in pyrite) or sulfur organically bound to carbon (as in dibenzothiophene). ΔH is defined as a small amount of energy in the form of activation energy required to induce sulfur gasification. Thus the in situ reactions allow sulfur to be liberated without transference of reaction elements to the carbonaceous aggregates.

3.2. Effect of leaching temperature and time

Peroxyacetic acid was used as solvent under the reaction conditions of 25–85 °C and 30–120 min in atmospheric pressure to study the extent of sulfur removal. The results are shown in Table 3. It can be seen with increasing of temperature from 25 to 55 °C the reduction of pyritic, organic and total sulfur increased.

Further increasing of temperature to 85 °C (just below boiling point) decreased the sulfur removal. It suggests temperature has a positive effect on desulfurization but because of decomposition of peroxyacetic acid on just below boiling point temperature, the reaction between hydroxyl cations and sulfur atoms decreased.

The sulfur removal increases sharply to 35.86% within 30 min and reaches to 61.36% on 90 min, but after 90 min it dose not changed significantly. It seems for this microwaved sample, the most of weak bound sulfurs eliminate within 90 min, after which the sulfur removal is relatively slow.

Since the pyritic, organic and total sulfurs are effectively removed and coal yield is reached to about 95%, So 55 °C and 90 min may be accepted as appropriate temperature and time, respectively.

3.3. Effect of particle size on acid leaching

To evaluation of particle size effect on chemical desulfurization process, the representative sample has divided to the fractions of 850–1400, 300–850 and <300 μm, microwaved (1000 W and 80 s) and then leached at 55 °C for 30–120 min with peroxyacetic acid. The results are shown in Table 4. It can be seen due to increasing in external surface area per unit mass of coal the rate of sulfur removal increased with decreasing of particle sizes from 850–1400 to <300 μm for both microwave and peroxyacetic acid washing processes. Because of fine distribution of pyrite in organic matrix the said results could be predicted with attention to Fig. 2. From this it must be concluded that in microwave irradiation stage, with decreasing of coal particle size, the absorption of microwave energy on sulfur-bearing areas or compounds to effect volatilization of sulfur to form of a stable gaseous compound have been increased. Also it can be seen there is mass transfer limitation in process and peroxyacetic acid has not free access to the reactive sites on the surface and also within particles. So these results indicate that grinding to small particle size is necessary to enhancing the level of desulfurization.

Table 3
Effects of leaching temperature and time on desulfurization of microwaved coal on 1000 W and 80 s (− 1400 μm)

Temperature (°C)	Time (min)	S content (db%)				S reduction (%)				Coal yield (%)
		Sulfate	Pyritic	Organic	Total	Sulfate	Pyritic	Organic	Total	
Microwaved Unleached coal	–	0.032	0.53	0.618	1.18	–	–	–	–	–
25	30	0	0.4	0.59	0.99	100	26.02	6.42	17.76	98
	60	0	0.32	0.52	0.84	100	41.05	17.86	30.5	97.62
	90	0	0.26	0.48	0.74	100	53.05	25.66	39.98	95.7
	120	0	0.15	0.45	0.60	100	73.08	30.75	51.64	95.1
55	30	0	0.27	0.51	0.78	100	50.56	19.92	35.86	97.03
	60	0	0.15	0.50	0.65	100	72.75	22.11	46.96	96.27
	90	0	0.06	0.42	0.48	100	89.24	35.45	61.36	94.97
	120	0	0.05	0.42	0.47	100	91.12	36.03	62.51	94.12
85	30	0	0.39	0.47	0.86	100	29.2	26.83	29.88	96.21
	60	0	0.32	0.48	0.8	100	41.89	25.25	34.75	96.23
	90	0	0.24	0.41	0.65	100	56.79	36.7	47.44	95.41
	120	0	0.22	0.42	0.64	100	60.89	35.97	48.9	94.21

3.4. *Effect of microwave irradiation on enhancing of desulfurization with peroxyacetic acid*

To study the effect of microwave irradiation on increasing of sulfur reduction by peroxyacetic acid, fraction size less than 300 μm, in two cases, microwaved and unmicrowaved, were leached with peroxyacetic acid at 55 °C for 90 min. The results are shown in Fig. 3. It can be seen after irradiation, the reduction on pyritic, organic and total sulfur increased from 49.86, 23.76 and 36 to 86.59, 35 and 61.89%, respectively.

It can be seen the effect of microwave pretreatment on reduction of inorganic sulfur is higher than organic one. It is result of higher dielectric constant of pyrite in comparison to its neighbor organic material.

Structural properties of coal as porosity, surface area and pore size play an important role in chemical desulfurization. Coal contains amounts of moisture in pores and this moisture is removed generally by heating around 100 °C. Borah and Baruah [21] have shown that oxidation of coal with heating, as a pretreatment process for coal desulfurization, decompose the big organic sulfur molecules to low molecular weight products so that these smaller molecules are much more prone to attack by the leaching solution in subsequent stages for the rupture of C–S as well as S–S bonds. Also they found that low temperature oxidation converts coal organic sulfur to compounds containing S=O and $-SO_2$ [22]. In these aerial oxidized samples, besides aliphatic sulfur, aromatic disulfide compounds can also be leached out [21].

Table 4
Effects of particle size on desulfurization of microwaved sample (1000 W and 80 s) with peroxyacetic acid at 55 °C

Particle size (μm)	Weight (%)	Time (min)	S content (db%)				S reduction (%)				Coal yield (%)
			Sulfate	Pyritic	Organic	Total	Sulfate	Pyritic	Organic	Total	
− 1400 + 850	17.46	Raw coal	0	0.68	0.51	1.19	–	–	–	–	–
		Microwaved Unleached	0.02	0.57	0.49	1.08	–	16.83	4.68	9.96	99.21
		30	0	0.43	0.49	0.92	100	26.52	2.6	17.02	97.4
		60	0	0.38	0.44	0.82	100	35.93	13.7	27.03	96.1
		90	0	0.29	0.46	0.75	100	51.2	9.96	33.39	95.91
		120	0	0.26	0.42	0.68	100	56.7	18.64	40.23	94.92
− 850 + 300	37.21	Raw coal	0	0.73	0.64	1.37	–	–	–	–	–
		Microwaved Unleached	0.03	0.52	0.58	1.13	–	29.41	10.19	18.26	99.09
		30	0	0.31	0.56	0.87	100	42.71	7.22	26.02	96.09
		60	0	0.2	0.48	0.68	100	63.28	21	42.56	95.45
		90	0	0.11	0.4	0.51	100	79.97	34.7	57.26	94.68
		120	0	0.09	0.39	0.48	100	83.71	36.72	60.02	94.1
− 300	45.33	Raw coal	0	0.9	0.82	1.72	–	–	–	–	–
		Microwaved unleached	0.05	0.63	0.65	1.33	–	30.78	21.61	23.54	98.88
		30	0	0.3	0.62	0.92	100	54.33	8.53	33.67	95.89
		60	0	0.12	0.61	0.73	100	82.05	11.56	48.28	94.23
		90	0	0.09	0.45	0.54	100	86.59	35.03	61.89	93.84
		120	0	0.07	0.43	0.50	100	89.66	38.45	65.02	93.03

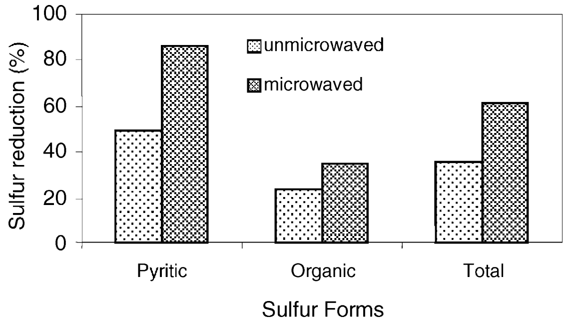

Fig. 3. Effect of microwave irradiation pretreatment on increasing of sulfur reduction with peroxyacetic acid at 55 °C for 90 min.

In this work it suggests that microwave irradiation pretreatment can remove moisture from coal pores with increasing of bulk coal temperature to 50–150 °C; so coal is more prone to attack by the peroxyacetic acid solvent in subsequent desulfurization stage.

3.5. FT-IR Spectroscopy of coal structure before and after desulfurization

Transformation taking places in microwaved/leached sample (< 1400 μm at 55 °C for 90 min) was studied by FT-IR spectroscopy. The samples were diluted with 85% by weight KBr and the spectra were recorded with a Bomer MB-100 instrument equipped with DTGS detector (Fig. 4).

The spectra were collected for each sample at a resolution of 4 cm^{-1} and analytical software provided by Bruker was used for spectral treatment. Intensity of the sharp and broad bands at 3619 and 3424 cm^{-1} interpreted as OH bonds that most probably is corresponding to OH groups bond to the mineral and organic matter, respectively. Intensity of the bands at 3043 and 3000–2700 cm^{-1} are corresponding to the stretching vibration of aromatic and aliphatic C–H bonds, respectively.

Coal consists of polycyclic materials. The bands observed at 1603 and 1437 cm^{-1} can be assigned to skeletal C–C stretching modes. Signals corresponding to organic sulfur is not visible in the spectra, while the bands in related to mineral matter (1200–1000 and 600–400 cm^{-1}) contained in coal are intensive. The minerals containing sulfur gave a band at about 425 cm^{-1} ascribed to pyrite [23].

As the spectra presented, the process of sulfur reduction carried out causes significant changes only in the peak corresponding to pyrite and the other mineral matter and organic matrix bands have not changed significantly. The result proves that this method on mild condition can use as secure process on sulfur reduction without destruction of coal organic matrix.

3.6. Technical considerations

Normally, high energy in a range of 600–900 °C is required to break bonded constituents of coal (i.e. Fe–S and C–S). In microwave desulfurization, energy absorbs at locations in coal where it is needed and sulfur can react

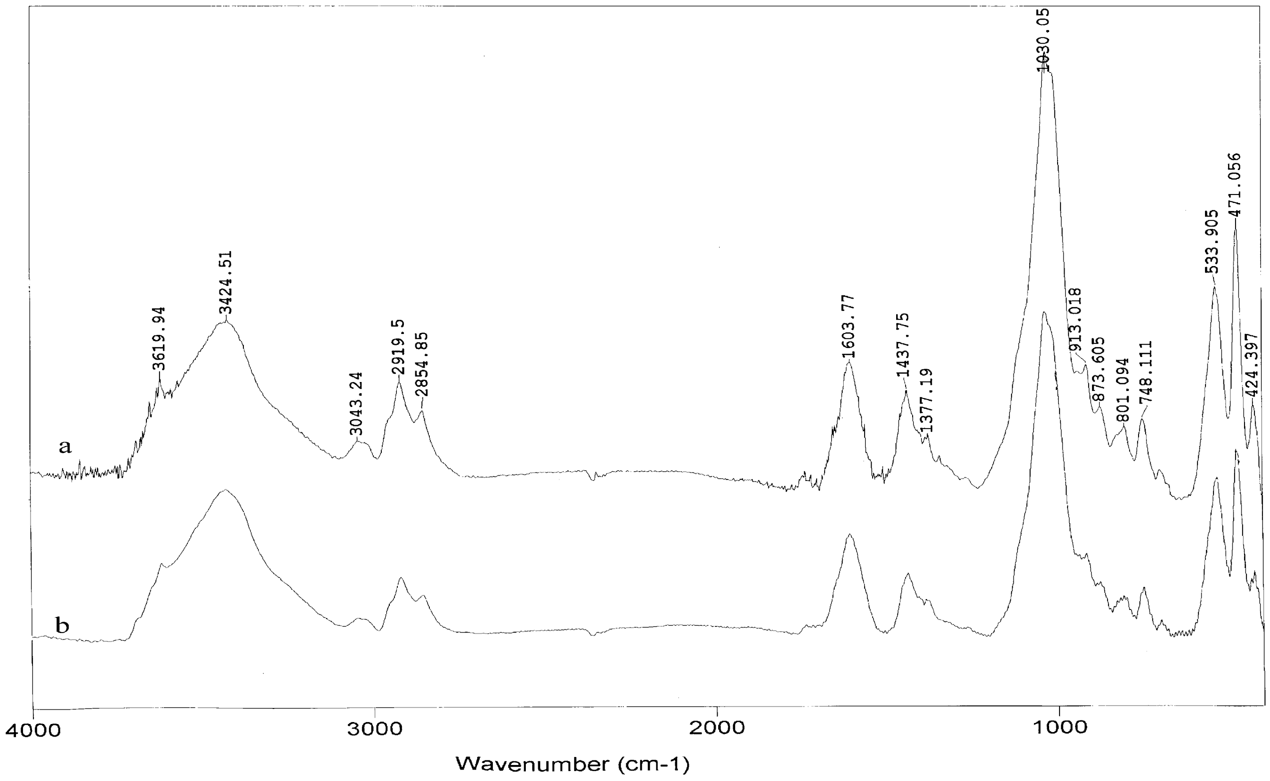

Fig. 4. FT-IR spectra of raw and microwaved/peroxyacetic acid washed samples: (a) raw coal; (b) microwaved/peroxyacetic acid washed.

with coal organic matrix rapidly than would be possible with conventional heating [12].

The heating energy generated by the microwave that is required to induce the in situ reaction is about 3 cal per gram. This is to be compared with 200 cal per gram required to heat the coal to around 800 °C for thermal removal of sulfur [12].

It should be noted that microwave heating is more expensive per unit of energy delivered than conventional heating. It is only beneficial if very rapid or selective heating is necessary.

In a previous investigation, Palmer et al. [15] used peroxyacetic acid for chemical desulfurization of coal at 50 °C for 6 h and achieved 56–63% reduction of total sulfur with coal yield of 78–83%. Also Sonmez et al. [16] examined desulfurization with peroxyacetic acid at 50 °C for 6 h and removed 36–75% of total sulfur with coal yield of 84%. In the present work, desulfurization of microwaved sample with peroxyacetic acid at 50 °C and 90 min removed about 62% of total sulfur with coal yield of 94%. This process also seems to reduce the organic sulfur in the coal matrix to a significant extent (35%). So desulfurization with microwave irradiation/ peroxyacetic acid washing should be commercially feasible in future because it does not require extra irradiation and leaching time and subsequently high energy demand; also leaching temperature is low and coal yield is higher than previous works.

In Iran, the acetic acid price is approximately 0.2 US$/l and the operating cost of coal cleaning process should be reduced considerably if acetic acid can be recovered and reused. This method should be possibly feasible in the country as regulations of SO_2 emissions are tightened and low-sulfur coal reserves are depleted. Further work should be undertaken to evaluate the overall economics of the process which will take account of the cost of microwave energy, coal drying and chemical reagents that a comparison can be made with other sulfur removal process.

4. Conclusion

Microwave irradiation and peroxyacetic acid washing combination is effective process for removal sulfur from coal. On optimized condition, the pyritic, organic and total sulfur decreased from initial values of 0.77, 0.67 and 1.44 to 0.06, 0.42 and 0.48%, respectively. The particle size reduction had a positive effect on efficiency of leaching process. It suggests that there is mass transfer limitation in process and Peroxyacetic acid has not free access to the sulfur atoms within coal organic matrix. The results suggest that microwave pretreatment is sufficiently effective for desulfurization of Tabas coal with peroxyacetic acid in mild conditions. The FT-IR spectra are shown decreasing in pyrite peak intensity whiles the organic matrix bands are not changed significantly.

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