



Effect of leaching high sulphur subbituminous coal by potassium hydroxide and acid on removal of mineral matter and sulphur[☆]

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Abstract

The effect of leaching coal samples from Boragolai and Ledo collieries of Makum coal fields, Assam, situated in north eastern region of India with potassium hydroxide solution alone at 95 and 150 °C as well as followed by mild acid on demineralization and desulphurization was investigated. Potassium hydroxide alone leads to 2–19% demineralization and 16–30% desulphurization of the coal samples at 95 °C. Demineralization of the coals decreases to 1–11 and desulphurization increases to 26–43% on increasing the temperature to 150 °C. The decrease in demineralization is due to increased precipitation of potassium aluminosilicates. Demineralization of the coal may be enhanced to 28–45 and 39–68% and desulphurization to 22–35 and 34–53% at 95 and 150 °C, respectively, by leaching the potassium hydroxide treated coal with 10% hydrochloric acid which decomposes the potassium aluminosilicates to certain extent. The treatment almost completely remove the inorganic and up to 37% organic sulphur from the coal samples.

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1. Introduction

High ash and high sulphur coals are unsuitable for efficient use in carbonization, combustion, gasification, liquefaction etc. purposes. Utilization of such coals leads to environmental pollution and other deleterious effects. The high quality coal reserves in the world are gradually depleting and, therefore, there is a growing interest to utilize the inferior grades of coal which contain high ash and sulphur. It is necessary to demineralize and desulphurize such coals prior to utilization.

There are large reserves of coal in Assam and other states in the north eastern region of India [1]. These coals are subbituminous in rank, characterized by high (2–7%) sulphur, high (30–50%) volatile matter and high (18–30) caking index (for the coking coals), high (6500–8000 kcal/kg) calorific value and low (1000–1050 °C) ash fusion temperature. Assam coal with low ash finds major use as a blend in metallurgical coke preparation for steel making. The coke for the purpose should have low (5–7%) ash and

low (maximum 0.7%) sulphur. The ash content of Assam coals is usually low (3–15%) but due to open cast and mechanical mining and winning of the coal from lower horizons, it is gradually increasing. The major minerals commonly found in the coals are quartz and other forms of silica, clay minerals, sulphides, carbonates, etc. The sulphur exists in the forms of sulphate, pyritic and organic [2–4] and about 70% of which is in the organic form. The high sulphur and ash content restrict large scale utilization of Assam coal.

Demineralization and desulphurization of coal can be achieved by both physical and chemical methods. The effectiveness of different methods for the purposes depends on the structure and composition of the minerals and their association in the coal. The physical methods are based on the differences in the physical properties of the minerals and the carbonaceous part of the coal. The chemical methods which involve treatment with different chemicals are effective for removing mineral matter, which are finely distributed and bound strongly to the coal.

Chemical cleaning of coal with alkali and acid solutions has been studied since long. Molten caustic leaching process is effective in reducing significant amounts of ash-forming minerals, pyritic sulphur and organic sulphur from coal [5–17]. Norton et al. [13] reported removal of 60–90% ash

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and sulphur from some bituminous coals from New Zealand using fused caustic. About 95% of the organic sulphur in the coal was in the form of complex thiophenes. Sulphur and ash removal was found to increase with increase of reaction time and temperature. Markuszewski et al. [11] reported removal of 80–90% of ash and 70–80% of total sulphur present in several bituminous coals with 80–90% recovery by treating with molten mixtures of NaOH and KOH at 350–370 °C. In TRW Gravimelt Process [7,9], molten mixtures of NaOH and KOH rather than NaOH alone desulphurize coal more efficiently. Kusakabe et al. [17] also reported that sulphur removal from coal samples depends on the KOH content in the NaOH and KOH mixtures used in leaching.

Leaching with aqueous solutions of caustic soda alone or followed by mild acid at a relatively low temperature and pressure also removes considerable amount of ash and sulphur from various coals. Araya et al. [18] achieved reduction of 29% ash and 30 wt% total sulphur from a subbituminous coal from Chile by treatment with 10% sodium hydroxide solution at 80 °C for 8 h. The ash and sulphur removal increase with increase of reaction time, temperature, alkali concentration and decrease of coal particle size. Kara and Ceylan [19] achieved removal of 60 wt% total sulphur and 65 wt% of the ash from some lignite samples of Turkey by treatment with 20% sodium hydroxide solution at 70 °C. Bolat et al. [20] achieved about 46% demineralization of a high ash-low sulphur bituminous coal from Turkey by treatment with 2% aqueous sodium hydroxide solution followed by 10% mineral acid under mild condition. Harada et al. [21] reported preparation of ultra clean coal from Taiheiyo coal (Hokkaido, Japan) by following aqueous caustic leaching method. Mukherjee and Borthakur [22] achieved removal of 43–50% of the ash, total inorganic sulphur and around 10% organic sulphur from Assam coal by treatment with 16% sodium hydroxide solution followed by 10% hydrochloric acid at 90–95 °C. Potassium hydroxide being more reactive than sodium hydroxide is expected to improve the efficiency of coal cleaning. The present communication reports the effect of aqueous solution of potassium hydroxide alone and followed by mild hydrochloric acid treatment on demineralization and desulphurization of high sulphur subbituminous Assam coal.

2. Experimental

Coal samples were collected from Boragolai and Ledo collieries of Makum coalfield, Assam, belonging to north eastern coalfields, India. The samples were stored under atmospheric conditions for few months and ground to below 212 µm fineness (72 B.S.) and preserved in airtight container.

The proximate analysis of the coal samples were done by following Indian Standard methods (IS:1350(part I)-1984).

The percentage of carbon, hydrogen and nitrogen were estimated by using a Perkin–Elmer (model 2400) elemental analyzer and total sulphur by following Eschka method (ASTM D 3177). The percentage of oxygen was calculated by difference. The forms of sulphur were determined by following (ASTM D 2492) methods. Ash analysis of the samples were done by standard chemical analysis methods [23,24]. The alkali metals were estimated using flame photometric method. For the purpose, the ash samples were fused using a mixture of NH₄Cl and CaCO₃ at 700 °C, and the fused mass after cooling was repeatedly extracted with water. The extract after removal of SO₄²⁻, Fe³⁺, Al³⁺, etc., were used for estimation of the alkali metals.

The coal was separately extracted with dilute HCl and dilute HNO₃. Sulphate sulphur was estimated in the HCl extract gravimetrically as BaSO₄ and non-pyritic iron was estimated from the same extract volumetrically using potassium dichromate as the oxidant. The total iron (pyritic and non-pyritic) was estimated from the nitric acid extract. The difference of the total and non-pyritic iron gives the amount of pyritic iron, from which the amount of pyritic sulphur was calculated. The organic sulphur was calculated from the difference of total and sum of pyritic and sulphate sulphur. The analyses were carried out in quadruplicate and the average values have been reported. The accuracy of the estimated values was ±0.01 for pyritic sulphur, ±0.01 for sulphate sulphur, ±0.02 for organic sulphur and ±0.02 for total sulphur. The proximate and ultimate analyses of the samples are presented in Table 1. The ash analysis of the coal samples are shown in Table 2.

The ground coal samples (about 10 g) were mixed with 50 ml potassium hydroxide solution of various concentrations and refluxed under atmospheric condition with stirring for 8 h at 95 °C. In some cases, the reaction mixtures were transferred to a steel autoclave lined with Teflon and heated at 150 °C (4.698 atm) for desired periods. The reaction mixtures were cooled, filtered and washed repeatedly with distilled water till alkali free. The samples were dried at 90 °C in an air oven and suspended in 50 ml

Table 1
Analysis of coal samples

	Boragolai	Ledo
<i>Proximate analysis (wt% as received)</i>		
Moisture	5.4	4.9
Ash	8.4	10.4
Volatile matter	41.4	41.5
Fixed carbon	44.8	43.2
<i>Ultimate analysis (wt% dry basis)</i>		
Carbon	68.8	70.0
Hydrogen	5.1	5.2
Sulphur	4.2	4.3
Nitrogen	1.5	1.4
Oxygen (by difference)	20.4	19.1
Calorific value (kcal/kg)	7527	7327

Table 2
Chemical analysis of coal ash

Source	Constituents (wt%)									
	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	CaO	MgO	Na ₂ O	K ₂ O	SO ₃	SAR	
Boragolai	57.9	18.9	11.9	3.9	4.7	0.1	1.4	0.8	3.06	
Ledo	54.8	21.8	12.7	2.6	3.8	0.2	1.2	1.3	2.51	

SAR (silica/alumina ratio).

10% hydrochloric acid solution. The mixtures were stirred under reflux for 8 h at 95 °C, filtered, washed and dried by adopting the same procedure as detailed above. The samples were analyzed for ash, and different forms of sulphur.

XRD patterns and FTIR spectra were used to characterize the mineral present in the coal. The XRD patterns were obtained using a Philips Analytical X-ray B.V. diffractometer. The FTIR spectra in the range of 4000–375 cm⁻¹ were recorded in KBr disc using FTIR 2000 Perkin–Elmer spectrophotometer.

3. Results and discussion

The chemical analysis (Table 2) shows that the major constituent of coal ash is silica (over 50%) followed by alumina and iron oxide. These three together constitute around 89% of the ash. Boragolai coal ash is more siliceous (SiO₂/Al₂O₃ wt ratio 3.06) than Ledo coal ash (SiO₂/Al₂O₃ wt ratio 2.51). XRD patterns and FTIR spectra (figures not shown) indicate presence of quartz as the major crystalline material in both the coal samples followed by clay minerals. The clay minerals identified are kaolinite, illite and chlorite. The coal samples also contain small to trace amounts of calcite, pyrite and marcasite. Ledo coal in addition contains gypsum and albite. The XRD patterns also reveal presence of amorphous material in both the coal samples. The high silica/alumina ratio in Boragolai coal ash suggests that the coal contains relatively high amount of free silica, which may be both crystalline (quartz) or amorphous.

The effect of leaching the coal samples with various reagents on ash removal is presented in Table 3. Demineralization of Ledo coal with KOH alone like that with NaOH [22] in general, is more than Boragolai coal and it increases with the concentration of the alkali. This is attributed to differences in the mineralogical composition of the coal samples. Ledo coal is less siliceous than Boragolai coal and it contain gypsum and albite as additional phases. Gypsum is highly soluble in alkali and the solubility of albite in alkali is also considerable [25]. Compared to NaOH, KOH leaching leads to more demineralization of the coal samples. KOH solution of 2 and 4%, respectively, remove 10 and 12.7% ash from Ledo and 6.8 and 5.7% ash from Boragolai coal at 95 °C. These values are higher than those achieved earlier with NaOH. Increase of temperature to 150 °C has little effect on demineralization of Ledo coal with 2% KOH, but it

Table 3
Effect of alkali/acid treatment on demineralization of the coal samples, temperature 95 °C, reaction time 8 h

Treatments	Boragolai coal		Ledo coal	
	Ash (%)	Degree of demineralization (%)	Ash (%)	Degree of demineralization (%)
Nil	8.8	–	11.0	–
10% HCl	6.3	28.4	8.8	20.0
2% KOH	8.2	6.8 (3.5)	9.9	10.0 (9.7)
2% KOH ^a	8.5	3.4	9.8	10.9
2% KOH	5.9	32.9 (40.6)	7.9	28.2 (40.3)
+ 10% HCl				
2% KOH	4.6	47.7	6.7	39.1
+ 10% HCl ^a				
4% KOH	8.3	5.7 (a)	9.6	12.7 (9.1)
4% KOH	5.5	37.5 (41.4)	7.4	32.7 (41.0)
+ 10% HCl				
8% KOH	8.4	4.5 (a)	9.4	14.5 (7.7)
8% KOH	5.3	39.8 (44.9)	6.8	38.2 (42.4)
+ 10% HCl				
16% KOH	8.6	2.3 (a)	8.9	19.1 (6.6)
16% KOH ^a	8.7	1.1	10.8	1.8
16% KOH	5.0	43.2 (49.9)	6.0	44.9 (43.9)
+ 10% HCl				
16% KOH	2.8	68.2	4.8	56.4
+ 10% HCl ^a				

Data under parenthesis with NaOH [22]; a: ash gain.

^a At reaction temperature 150 °C.

substantially lowers demineralization of Boragolai coal. Demineralization of Ledo coal increases to 19.1% and Boragolai coal decreases to 2.3% at 95 °C on increasing the alkali concentration to 16%. Demineralization of both the coal samples at 150 °C decreases on increasing the alkali concentration from 2 to 16%. Alkali concentration and temperature are kinetic factors on mineral dissolution. The decreased demineralization of the coal samples with increase of these factors indicate that mineral dissolution reactions are followed by precipitation of some substance [26].

Potassium hydroxide extracts of the coal samples (shown in Table 4) contain SiO₂, Al₂O₃, Fe₂O₃, Ca²⁺, Mg²⁺, SO₄²⁻, etc., constituents. This is attributed to dissolution of water soluble materials like sulphates of calcium, magnesium

Table 4
Dissolved constituents of coal extracted with KOH at 150 °C (g/100 g of coal)

	Alkali concentration			
	Boragolai coal		Ledo coal	
	2%	16%	2%	16%
SiO ₂	1.12	1.46	1.14	2.60
Al ₂ O ₃	1.04	1.16	1.11	1.22
Fe ₂ O ₃	2.91	3.52	3.80	3.92
SO ₃	0.25	0.45	0.60	0.79
CaO	1.00	1.30	0.80	0.80
MgO	1.20	1.40	1.20	1.40

present in the coal or conversion of some water insoluble components to soluble salts due to reaction with KOH. Alkali reacts with sulphides, and materials containing SiO₂, Al₂O₃, Fe₂O₃, etc., acidic oxides (like amorphous silica, quartz, clay minerals, etc.) forming soluble sulphates, alkali silicates, aluminates, ferrates, etc. The solubility of these constituents changes with the nature and composition of the parent material. Amorphous silica dissolves more than crystalline silica and the solubility of the clay minerals are also different [27]. Dissolved silicates and aluminates may interact and form sparingly soluble alkali-aluminosilicate hydrogel. Potassium aluminosilicate gel may precipitate when the concentration of the dissolved potassium silicate and potassium aluminate ions exceed the solubility product of potassium aluminosilicate [28].

The amount and composition of precipitated potassium aluminosilicates depend on the initial composition of the solution. The data in Table 4 shows that the content of each element in the extracts of both the coal with 16% KOH solution is higher than that with 2% solution, but the degree of demineralization of the coal samples is less with the 16% KOH solution than the 2% solution (Table 3). This is attributed to increased formation of insoluble potassium aluminosilicates and their accumulation in the coal samples when leached with 16% KOH solution. Many earlier workers while investigating with sodium hydroxide also observed decrease in demineralization of several coal samples with increased concentration of the alkali and attributed the same to formation of insoluble sodium complex like zeolites, feldspaths, hydroxy sodalite, hydroxy cancrinite, etc. [29–31].

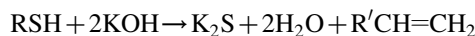
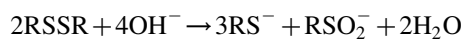
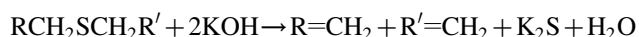
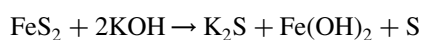
Demineralization improves significantly when the KOH treated coal samples are treated further with mild (10%) hydrochloric acid, which cause decomposition of many oxides, sulphides and carbonates present in the coal and potassium aluminosilicates formed in the first step forming soluble salts. Demineralization of the coal samples by the two step process (alkali followed by acid) involving KOH, however, is not as high as found with NaOH, in spite of the higher demineralizing ability of KOH than NaOH. This is attributed to incomplete decomposition of potassium aluminosilicates formed in the first step by the mild acid used in the treatment. This is reflected from the potassium content of some of the treated samples shown in Table 5. The improvement, like those found in NaOH leaching depends on the concentration of the KOH and reaction temperature used in the first step. Demineralization of Boragolai and Ledo coal by the two step process increases, respectively, from about 33 and 28% to 43 and 45% at 95 °C and from about 48 and 39% to 68 and 56% at 150 °C on the same increase of the KOH concentration from 2 to 16%.

Desulphurization of the coal samples increases with increase of KOH concentration (Table 6) and it is attributed to solubilization of the sulphates, and conversion of pyritic and some organic functional groups like thiols, disulphide, etc., present in the coal to soluble salts [32]. Some schematic

Table 5
Potassium content in some treated coal samples

Treatment	Potassium content (K ₂ O%)
<i>Boragolai coal</i>	
16% KOH at 95 °C	6.0
16% KOH at 95 °C followed by 10% HCl	2.2
16% KOH at 150 °C	7.5
16% KOH at 150 °C followed by 10% HCl	3.3
<i>Ledo coal</i>	
16% KOH at 95 °C	5.5
16% KOH at 95 °C followed by 10% HCl	2.6
16% KOH at 150 °C	6.8
16% KOH at 150 °C followed by 10% HCl	3.4

reactions are shown below



Potassium hydroxide of 2% at 95 °C removes over 24 and 16% of the total sulphur present in Boragolai and Ledo coal. The total sulphur removal increases to around 30 and 25%, respectively, on increasing the alkali concentration to 16%. These values are much higher than those (21 and 19%) achieved with sodium hydroxide solution from the same

Table 6
Effect of alkali/acid treatment on desulphurization of the coal samples, temperature 95 °C, reaction time 8 h

Treatments	Boragolai coal		Ledo coal	
	Total sulphur (%)	Degree of desulphurization (%)	Total sulphur (%)	Degree of desulphurization (%)
Nil	4.27	–	4.31	–
10% HCl	3.43	19.7	3.85	10.7
2% KOH	3.22	24.6 (18.3)	3.61	16.2 (13.7)
2% KOH ^a	3.15	26.2	3.00	30.4
2% KOH + 10% HCl	3.12	26.9 (25.1)	3.35	22.3 (20.6)
2% KOH + 10% HCl ^a	2.52	40.9	2.83	34.3
4% KOH	3.16	25.9 (19.0)	3.52	18.3 (14.6)
4% KOH + 10% HCl	3.04	28.8 (26.2)	3.20	25.7 (23.9)
8% KOH	3.05	28.6 (19.4)	3.45	19.9 (15.5)
8% KOH + 10% HCl	2.93	31.4 (29.3)	3.12	27.6 (26.7)
16% KOH	2.98	30.2 (20.6)	3.23	25.1 (19.2)
16% KOH ^a	2.42	43.3	2.70	37.3
16% KOH + 10% HCl	2.76	35.4 (33.2)	2.86	33.6 (29.9)
16% KOH + 10% HCl ^a	2.02	52.7	2.12	50.8

Data under parenthesis with NaOH [22].

^a At reaction temperature 150 °C.

Table 7
Effect of KOH concentration (followed by 10% HCl) on distribution of sulphur in the coal samples

KOH Conc. (%)	Sulphur distribution (wt%)					
	Boragolai coal			Ledo coal		
	Pyrite	Sulphate	Organic	Pyrite	Sulphate	Organic
a	0.64	0.52	3.11	0.52	0.41	3.38
2	0.04 (0.10)	0.03 (0.05)	3.05 (3.05)	0.03 (0.05)	0.02 (0.03)	3.30 (3.34)
4	0.02 (0.08)	0.01 (0.03)	3.01 (3.04)	0.02 (0.03)	0.01 (0.01)	3.17 (3.24)
8	0.00 (0.06)	0.00 (0.01)	2.93 (2.95)	0.01 (0.02)	0.00 (0.01)	3.11 (3.13)
16	0.00 (0.02)	0.00 (0.00)	2.76 (2.83)	0.00 (0.00)	0.00 (0.00)	2.86 (3.02)
2 ^a	0.02	0.01	2.49	0.01	0.00	2.82
16 ^a	0.00	0.00	2.02	0.00	0.00	2.12

Data under parenthesis with NaOH [22]; a: values for unleached coal.

^a At reaction temperature 150 °C.

coal samples under similar conditions [22]. The sulphur removal from the coal samples using 2 and 16% KOH increases to about 26–43% and 30–37%, respectively, by increasing the reaction temperature to 150 °C.

Desulphurization, like that of demineralization improves on treatment of the KOH treated coal samples with mild hydrochloric acid. Desulphurization by the two step process also depends on the concentration and temperature of KOH used in the first step. Increase of concentration of KOH from 2 to 16% increases desulphurization of Boragolai and Ledo coals from about 27 to 35% and 22 to 34% at 95 °C and from about 41 to 53% and 34 to 51%, respectively, at 150 °C. Desulphurization using KOH by the two step process is marginally higher than using NaOH although the former possesses much higher desulphurization ability than the later. This is also attributed to higher acid stability of potassium aluminosilicates than sodium aluminosilicates. Under the same condition, sodium aluminosilicates and potassium aluminosilicates may crystallize to products having different properties [33]. The aluminosilicates have the ability to entrap various salts and many crystalline aluminosilicates contain sulphur in the structure [34].

The sulphur distribution in some of the coal samples treated successively with alkali and acid is presented in Table 7 along with those achieved with NaOH under the same conditions [22]. Results show that KOH solution is slightly more effective in removal of different forms of sulphur than NaOH solution. Potassium hydroxide solution of 2% at 95 °C removes around 94% pyritic sulphur and 94–95% sulphate sulphur from the coal samples against removal of 84–90% pyritic sulphur and 90–92% sulphate sulphur by sodium hydroxide solution under similar conditions. The entire sulphate sulphur and over 98% pyritic sulphur from the coal samples may be removed by leaching with 8% KOH solution at 95 °C. Potassium hydroxide of 16% solution removes 11–15% organic sulphur against 9–11% as found with sodium hydroxide solution at 95 °C. Organic sulphur removal with the KOH solution increases to 35–37% on increasing the temperature

to 150 °C. The removal of high amount of organic sulphur is possibly due to low coalification rank of the coal [35–37]. These coals contain more of the organic sulphur in aliphatic or labile form, mainly as thio ethers (sulphides) and disulphide [38]. Sulphur distribution in Assam coal revealed presence of mercaptan, disulphide, thiol, sulphide and simple thiophene as the major functionalities [39]. The strong alkali probably removes the aliphatic sulphur and some simple heterocyclic compounds present in the coal.

4. Conclusions

Considerable amount of ash and sulphur can be removed from Assam coal by treatment with aqueous solution of potassium hydroxide alone or followed by mild hydrochloric acid. Potassium hydroxide alone removes 2–19% ash and 16–30% of the total sulphur from Boragolai and Ledo coal which increases, respectively, to 28–45% and 22–35% by the two step process. The acid treatment does not completely decompose the potassium aluminosilicate formed during the alkali treatment. Temperature and KOH concentration are kinetic factors on demineralization and desulphurization. The two step process almost completely remove the inorganic sulphur and 11–15% organic sulphur from the coal samples at 95 °C and the later increases to about 35–37% at 150 °C. The removal of relatively high amount of organic sulphur is attributed to low coalification rank of the coal where most of the sulphur exists as aliphatic or labile form.

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