

Semi-fused Salt-Caustic Mixture Leaching of Turkish Lignites - Sorel Cement Use for Desulfurization

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ABSTRACT: Beneficiate from clean fuels, particularly from clean coals, in industrial combustion systems and power stations for energy production is much significantly preferred in our country as well as in the world. Instead of beneficiation from high quality coals due to lack of the deposits, production of low quality coals and consumption in our country may create some the environmental issues. Low-grade coals with high ash and sulfur should be processed by the physical and chemical methods and later beneficiated. It has been fundamentally needed that the use of most feasible chemical methods for desulfurization and deashing of the Turkish lignites and hard coals since they might not be cleaned by physical methods. In this research, the one commonly used among various chemical methods, semi-fused salt-caustic mixture leaching of several Turkish lignites, for desulfurization and deashing was investigated and discussed for use marble waste fine and sorel cement in salt leaching of Turkish lignites. In this study, semi-fused salt-caustic leaching tests of different types of Turkish run-off-mine lignites; Aydın Şahinalı, Kütahya Gediz, Denizli Çivril, Bolu Mengen. Tunçbilek, Soma Kısırakdere were carried out. Optimum semi-fused leaching conditions and effects of different types of salts were investigated. Magnesia and Sorel cement, NaCl, MgCl₂ and KCl were also used as desulfurizing salt agent. Use of Sorel cement (3MgO.MgCl₂) -caustic mixture in coal desulfurization were distinctly investigated and discussed. The qualities of processed coal products are ascertained by chemical and standard coal analysis of ASTM 3174-3177. According to the test results, the best semi-fused salt-caustic leaching process and salt mixture was determined.

Keywords: Coal Desulfurization, Semi-fused Caustic Leaching, Salt Leaching

INTRODUCTION

Significant use of coal resources in the developed countries in primary energy production as a great alternative source lack of oil and natural gas resulted in great environmental issues, especially great amount of combustion gas evolution, even high carbon emissions. Low quality coal resources should be utilized by few successive cleaning and treatment processes following extraction. At legislative environmental concerns in Turkey, utilization of high sulfur Turkish lignites containing over 2% combustible sulfur and high ash Turkish coals containing over 10% ash have been restricted by local controllers. In Turkey approximately 80% (28 million tons) of total lignite production of 35 million tons was utilized in primary energy production.(Anonym, 2009)

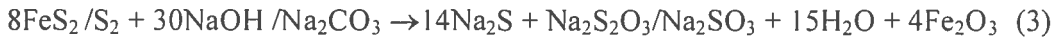
The methods using gravity and surface characteristics of different types of coals might decrease ash amount to a certain levels, but sulfur contents still remained with clean coal products (Tosun et al, 1994; Özbayoğlu ve Mamurekli, 2002). Biological processing of coal specimens has also been tested, and resulted in great amount of sulfur reductions obtained from high sulfur coals (Gürü et al, 2006; Bayram et al, 2002; Durusoy et al, 1992; Aytar et al 2008). Biological medium protection and environmentally safety procedures have needed still preferences of great concerns in the region (Tosun et al, 1994).

Flue gases of coal combustion were greatly researched for desulfurization in different combustion chambers and burning systems by different methods succeeding great desulfurization. The adsorption techniques and also different types of sorbents widely used in the studies (Demirbaş and Balat, 2004; Wheelock, 1979; Qi et al, 2004; Karaca, 2003; Gürü et al, 2008) and sulfur has been captured as sulfite and sulfate solutions or in solid forms of mixtures with reactive sorbents (Tosun et al, 1996, Tosun, 1996; Tosun, 1997; Tosun , 2007; Karatepe, 2000; Rongfang et al, 2007; Garcia and Moinela, 1991). 7% lime addition to coal during combustion reduced combustible sulfur emissions to certain levels (Ozbas et al, 2002; Altun et al, 2006).

The use of chemical methods, leaching by such as acids, ferric chloride, in coal desulfurization might decrease significant amount of sulfur contents (Rubio and Mastral 1989), but could not be much sufficient. It was determined that 0.3 M nitric acid solution reduced both combustible and total sulfur contents of Askale coal samples to 1.1 % sulfur levels (Gürü, 2007). Ferric iron leaching of Anatolian asphaltites were investigated and sulfur contents were significantly reduced (Hamamci et al, 1997). Autoclaved caustic leaching of coal samples in the solutions diluted 10 % caustic treated 4-6 hours reduced inorganic sulfur contents of coal samples, however organic sulfur still remained with coal samples and also surface characteristics of coal samples deteriorated (Yoon, 1991; Tosun and Çiçek, 1997).

Molten caustic leaching significantly reduced sulfur contents of coal samples and also organic sulfur content reduced to a lower level without destroying physical qualities of coals. Advantageous of molten caustic leaching are as follows:

- Reagents used in molten caustic leaching process are commonly caustic soda, caustic ash and very cheap alkalis widely available in the country and recycled in the process, or recovered,
- Sulfur contents of coals could be reduced lower levels, due to organic sulfur reacted,
- Product clean coal fine physical properties may not be changed,
- Thermo-briquetting technique of coals may be applied following the desulfurization treatment,
- More environmentally friend coal products could be obtained,
- Reduction of ash and volatile matter may produce richer carbon based semi-coke product by lost of moisture, nitrogen and carbon dioxide. Molten caustic mixtures especially including different type of chemical alkaline react with coal samples in reactors at atmospheric pressure by the equations as given below (Kumar et al, 2000);



It is also determined that the optimized alkali-acid leaching method of different type of low grade coals were managed in reduction of highly significant amount of ash and sulfur contents (Sharma and Gihar, 1991; Nabeel et al, 2009, Chriswell et al, 1994). Coal slurries were leached by caustic soda at 220-250°C and followed by acid liquors resulting in ultra clean coal products containing 0,2 % ash and similar low sulfur contents (Brooks et al, 2006; Culfaz et al, 1996; Mukherjee et al, 2003).

In this study, semi-fused salt-caustic leaching tests of different types of Turkish run-off-mine lignites; Aydın Şahinalı, Kütahya Gediz, Denizli Çivril, Bolu Mengen, Tunçbilek, Soma Kısırakdere were carried out. Lime, Hydrated lime, Magnesia, Sorel cement, NaCl, MgCl₂ and KCl were used as desulfurizing salt agent. Sorel cement -caustic mixtures use in coal desulfurization were virtually investigated and discussed.

MATERIALS AND METHOD

In this research, representative specimens of the different types of Turkish lignites; Aydın Sahinalı, Kütahya Gediz, Denizli Çivril, Bolu Mengen, Tunçbilek, Soma Kısırakdere were crushed and comminuted to minus 1mm size by controlled screening. Air dried samples of 40-50 gr from each different coal types were prepared and sealed in nylon bags. The results of proximate and ultimate analyses of various Turkish lignites used in the experiments are given in Table 1 and Table 2. The qualities of processed coal products are ascertained by chemical and standard coal analysis of ASTM 3173-3177.

Table 1. The Result of Proximate Analyses of Turkish Lignites.(ADB:Air dried base. DB:Dried base, DAF:Dried ashless base)

Coal Type	Ash,% ADB	Moisture,% ADB	Total S,% DB	Volatile Matter,% DAF
Aydın Şahinali	29.3	8.1	1.1	52.6
Kütahya Gediz	22.0	11.7	3.6	42.7
Denizli Çivril	15.2	12.7	3.0	45.2
Tunçbilek	25.7	5.7	3.9	38.2
Soma Kırakdere	13.8	14.0	2.2	40.4
Bolu Mengen	35.1	15.3	4.1	67.3

Table 2. The Result of Ultimate Analyses of Turkish Lignites. (DAF:dried ashless base.)

Coal Type	C,%	H,%	N,%	O,%	Calorific Value,MJ/kg
Aydın Şahinali	69.3	6.1	2.1	14.6	14,8
Kütahya Gediz	72.0	6.7	2.2	13.7	16.5
Denizli Çivril	70.2	6.7	2.1	14.2	17.2
Tunçbilek	77.7	4.9	2.1	10.2	19.5
Soma Kırakdere	78.8	4.3	2.0	10.4	19.8
Bolu Mengen	62.1	8.3	1.9	15.3	12,4

Screen analysis of Turkish lignite samples were made by standard Tyler Screens and particle size distributions and normal distributions of lignites samples are respectively illustrated in Figure 1.

Mixing ability of Turkish lignites may not be destroyed by controlled crushing and screening till reducing particle size of specimens to minus 1 mm. As seen from Figure 1, Specific surface area of coal samples was determined by benchmarked Rigden flowmeter and highly sufficient, over 950 cm²/gr, in order to react with semi-fused salt-caustic mixtures. As seen from Figure 1, 80% of weights of samples were 0,3 mm. Coal particles in lignite samples were mainly distributed between 100 and 300 µm size fractions. Higher specific surface area of 1160 cm²/gr for lignite samples was determined and also highly sufficient in order to react with semi-fused salt-caustics. Two normal distributions are seen from Figure 2 due to two different mechanical breakage manners of lignite samples and hard lignite maceral groups containing ash minerals. Especially, hardly crushed particle size fraction of lignite samples was ranging between 700 and 900 micron.

Semi-fused salt-caustic leaching experiments are carried out in a benchmarked laboratory type 1lt reactor put in sand furnace at atmospheric pressure at a temperature precision of ±5°C as seen from Figure 2.

Semi-fused salt leaching tests were carried out under atmospheric pressure at a constant time period of 30 minutes previously determined over 40-50 gr coal and lignite samples. Total salt-caustic mixture weight was hold constant at ½ of coal weight. Various salts were mixed at equal weight rate into caustic and added to coal samples.

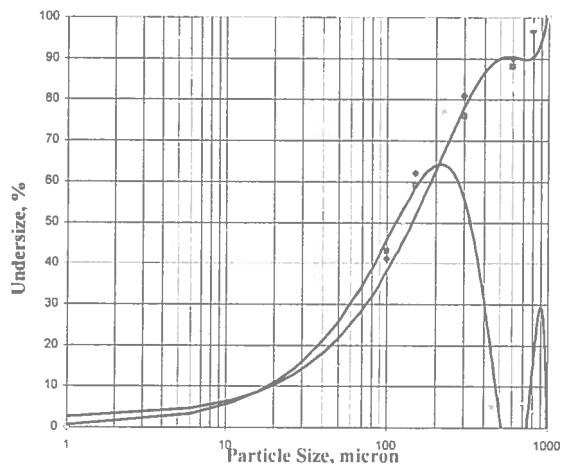


Figure 1. Particle Size and Normal Size Distribution of Turkish Lignites.

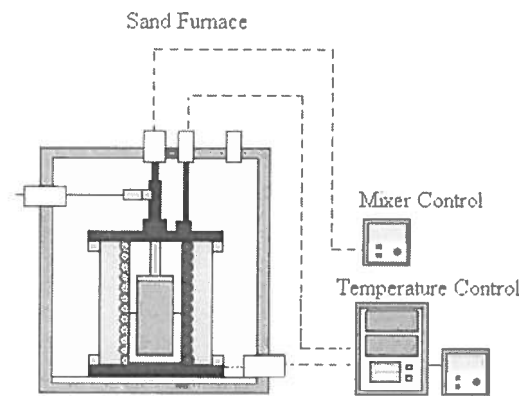


Figure 2. Semi-Fused Salt-Caustic Leaching Equipment.

RESULTS AND DISCUSSIONS

In this semi-fused salt-caustic leaching experiments, furnace temperature was hold between 100°C and 400°C and lignite samples were mixed only by unmelting lime and caustic soda mixture at ½ of coal weight. Produced coal specimens were washed, filtered and subjected to chemical analysis for sulfur hold-up determination. Test results of semi-fused salt-caustic leaching of Turkish lignite samples by only lime -caustic soda mixture addition are seen from Figure 3.

As shown in Figure 3, higher sulfur hold-up ratios by lime and caustic soda mixture just occurred between 250 and 400 °C leaching temperatures. However, sulfur hold-up ratios remained at low levels, and reached only 34-35% values for Soma and Aydın Lignite samples, respectively. Those high sulfur hold-up values were reached at the leaching temperatures ranging 300-350 °C. Total sulfur reduction of Turkish lignites was not sufficient in lime-caustic mixture leaching. Higher temperatures over 350 °C decreased desulfurization rate due to high volatile lost from coal samples. Lime might not be dissolved in melted caustic soda mixture in the concern of non penetrating and lower reacting by coal surfaces so that resulted in lower desulfurization rates.

In the next leaching experiments, furnace temperature was changed between 100°C and 400°C and lignite samples were mixed only by unmelting hydrated lime and caustic soda mixture at ½ of coal weight. Produced coal specimens were washed, filtered and subjected to chemical analysis for sulfur hold-up determination. Test results of semi-fused salt-caustic leaching of Turkish lignite leaching by hydrated lime and caustic mixture addition are seen from Figure 4.

As shown in Figure 4, higher sulfur hold-up ratios by hydrated lime and caustic soda mixture just occurred between 300-400 °C leaching temperatures. However, sulfur hold-up ratios reached only 68-72% values for Aydın Şahinali Lignite and Soma Lignite at 350 °C, respectively. Higher sulfur hold-up values were obtained at leaching temperatures ranging from 300 to 370 °C.

In the semi-fused salt-caustic leaching experiments with magnesia, furnace temperature was changed between 100°C and 400°C and lignite samples were mixed only by unmelting magnesia and caustic soda mixture at ½ of coal weight. Produced coal specimens were washed, filtered and subjected to chemical analysis for sulfur hold-up determination. Test results of semi-fused salt-caustic leaching of Turkish lignites by magnesia and caustic mixture addition are seen from Figure 5.

As shown in Figure 5. higher sulfur hold-up ratios by magnesia and caustic soda and ash mixture just occurred between 250°C and 400°C leaching temperatures. However, sulfur hold-up ratios reached only 75%, 74% and 76% values for Soma, Kütahya Gediz and Aydın Lignites, respectively. Those hold-up

values were obtained at leaching temperatures of 300 and reduced slightly at 370 °C. Bolu Mengen and Denizli Çivril lignites could be desulfurized at lower levels of 46% and 44%, respectively, due to higher amount of clay mineral fines covering pores and micro fissures in the lignite coal particles. Higher pore volumes of Bolu Mengen and Denizli Çivril lignites at lower rank caused decreased desulfurization rates in the salt-caustic leaching.

In the semi-fused salt-caustic leaching experiments with Sorel Cement, furnace temperature was changed between 100°C and 400°C and lignite samples were mixed only by unmelting sorel cement(containing 3 part MgO to 1 part Mg chloride) and caustic soda mixture at ½ of coal weight rate. Produced coal specimens were washed, filtered and subjected to chemical analysis for sulfur hold-up determination. Test results of semi-fused salt-caustic leaching of Turkish Lignites by sorel cement caustic mixture addition are seen from Figure 6.

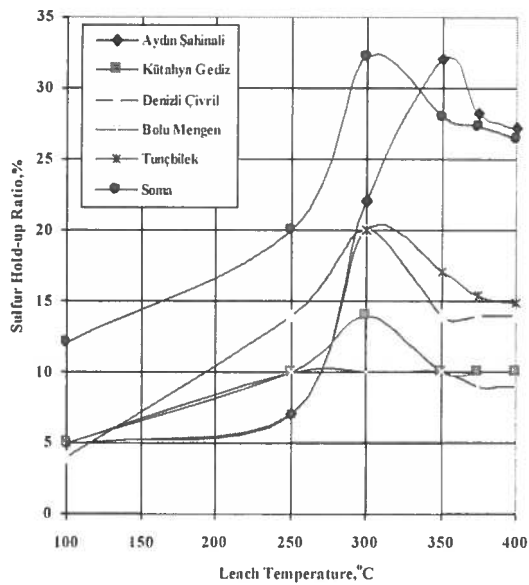


Figure 3. Sulfur Hold-up Rates in fused Caustic Leaching of Turkish Lignites by Lime and Caustic Soda.

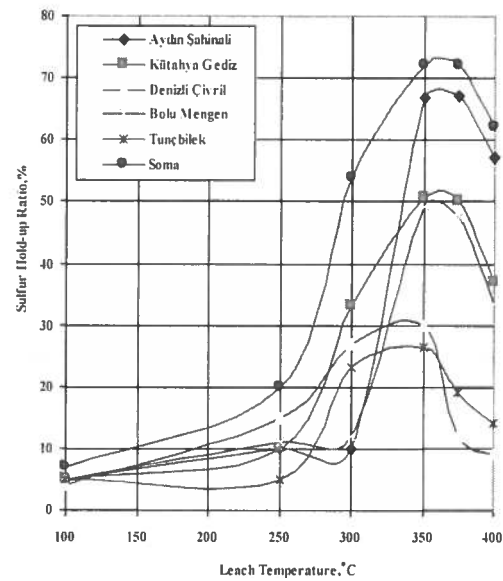


Figure 4. Sulfur Hold-up Rates in Semi-fused Salt-Caustic Leaching of Turkish Lignites by Hydrated Lime and Caustic soda.

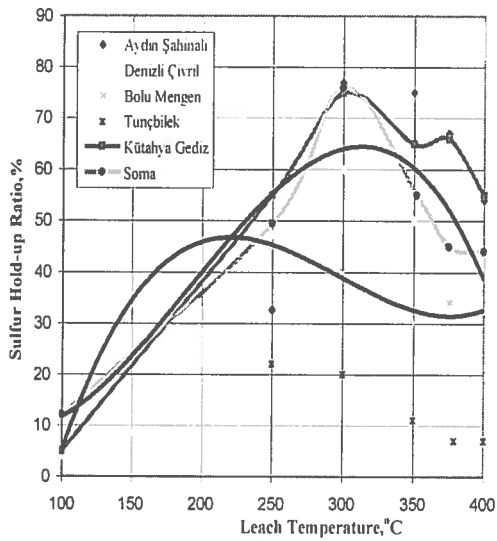


Figure 5. Sulfur Hold-up Rates in Semi-fused Salt-Caustic Leaching of Turkish Lignites by Magnesia and Caustic soda.

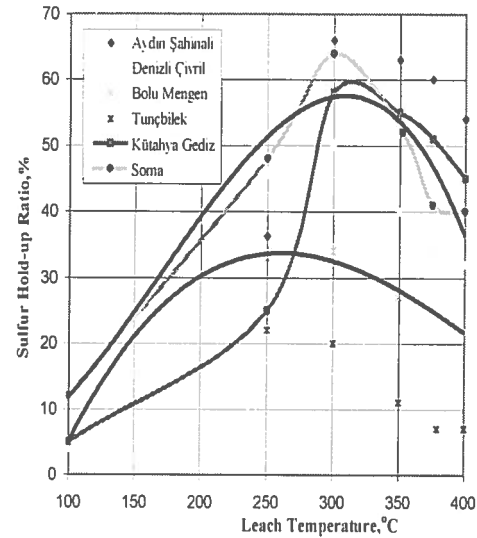


Figure 6. Sulfur Hold-up Rates in Semi-fused Salt-Caustic Leaching of Turkish Lignites by Sorel Cement and Caustic soda.

As shown in Figure 6, higher sulfur hold-up ratios by sorel cement and caustic soda mixture just occurred between 250 °C and 400 °C leaching temperatures. However, sulfur hold-up ratios reached only 68-75% values for Soma and Aydın Lignites at 300 °C, respectively. Those higher hold-up values were obtained at leaching temperature of 300 °C. 70% magnesia content of sorel cement resulting from mixture of magnesium chloride and magnesia was greatly beneficial in desulfurization of lignites.

In the following semi-fused salt-caustic leaching experiments, furnace temperature was kept constant at 300 and 350°C and lignite samples were mixed only by unmelting magnesia, hydrated lime Sorel cement and NaCl, MgCl₂ and KCl (melting around 800 °C, just salt hydrates may fuse) with caustic soda at ½ of coal weight. Leached coal specimens were washed, filtered and subjected to chemical analysis for sulfur hold-up determination. Test results of semi-fused salt-caustic leaching of Turkish lignites by various salts are seen from Figure 7.

As shown in Figure 7, higher sulfur hold-up ratios by hydrated lime, magnesia and Sorel Cement- caustic mixtures occurred at 300 °C and comprising also 70% magnesia content reduced significant amount sulfur at 300 °C leaching temperatures. However, sulfur hold-up ratios reached over approximately 73% level values for Soma and Aydın Lignites by hydrated lime use in leaching, respectively. However, lower sulfur hold-up values were obtained for other unmelted salt mixtures such as NaCl, MgCl₂ and KCl due to acidic and chloride acid vapors non-reacting over coal surfaces and pores instead of activation in the leaching temperature of 300 and 350 °C.

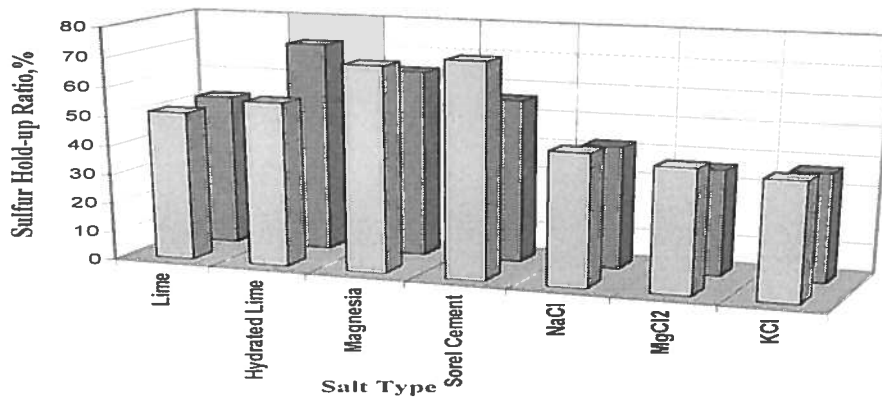


Figure 7. Sulfur Hold-up Rates in Semi-fused Salt-Caustic Leaching of Soma and Aydın Lignites by Various types of Salts and Marble waste fine-Caustic Soda Mixtures at 300 and 350°C, respectively (light and dark).

As shown in Figure 8, volatile matter values of Turkish lignites were decreased to lower 25-40 % in general range by physical and chemical deterioration of coal particles over 350 and 400 °C by semi-pyrolysis with lack of oxygen content in the reactor. Higher volatile matter reductions occurred between 350 °C and 400 °C leaching temperatures. However, highest volatile matter reduction and physical and chemical deterioration occurred at Bolu Mengen, Denizli Çivril and Aydın Lignites due to higher volatile contents and highly reactive and burning abilities at 400 °C leaching temperatures. As a result of TGA analysis of Turkish lignites, the coals slightly lost less amount of volatile matter between 200 and 350 °C due to lower original volatile matters. As a result of those volatile reduction percents, it is distinctly told that semi-pyrolysis of Turkish lignites might occur in semi-fused salt-caustic leaching of Turkish lignites. However, Bolu Mengen and Denizli Çivril lignite might loose higher volatile matter compared with other type of lignites.

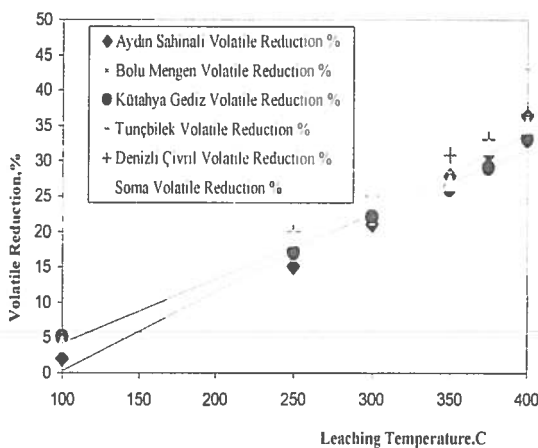


Figure 8. Volatile matter reduction ratios in Semi-fused Salt-Caustic Leaching of Turkish Lignites by Sorel Cement and Caustic Mixture.

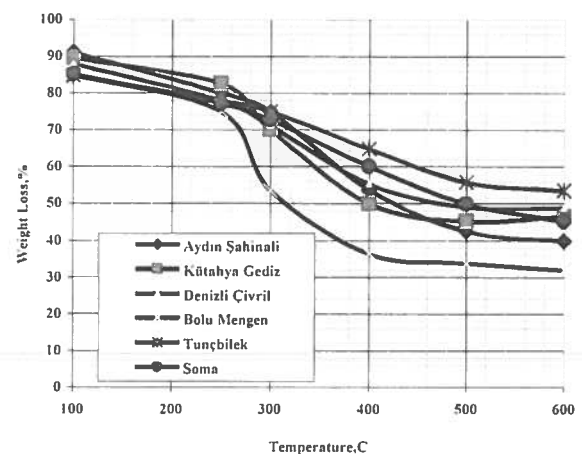


Figure 9. TGA Weight Loss of Turkish Lignites

CONCLUSIONS

In this study, it is mainly concluded those as follows;

At furnace temperature changed between 300 and 350°C semi-fused leaching was sufficiently managed. Over 350°C may deteriorate physical and chemical features of lignite samples by commence of pyrolysis.

Semi-fused salt caustic leaching of different types of Turkish lignites was successfully processed in terms of desulfurization and even reduction of volatile matter. Addition hydrated lime with caustic soda over Turkish lignite samples in semi fused leaching at furnace temperature changed between 300 to 350°C was proposed for desulfurization and even devolatilization.

Aydın Sahinali and Soma lignites were successfully desulfurized at 73-60% rate levels due to low ash and high reactive sulfur contents in coal structure. Kutahya Gediz and Bolu Mengen lignites were partially desulfurized at 51-49% rate levels due to higher volume percents of pore structure at lower ranks.

Sorel cement may be sufficiently used in desulfurization instead of magnesia and hydrated lime. It is virtually concluded that greater desulfurization rate of 76% was obtained from Soma and Aydın lignite at 300°C and also proposed in use as sorbent agent in pelletized or briquetted coal products. Magnesia contents of Sorel cement during leaching may be hydrated and cover sulfur components. It was also distinctly ascertained that sorel cement containing approximately 70% magnesia and magnesium chloride was efficient as a desulfurizing sorbent in the caustic mixtures.

Especially, other salt chemicals, surfactants and ash powders may also be used in semi-fused salt-caustic leaching without deteriorating coal chemical structure.

It was distinctly determined that semi-pyrolysis of leached samples occurred at approximately 20-25% rate regarding to devolatilization ability of lignite samples at lower temperatures.

According to the test results, the best semi-fused salt-caustic leaching process conditions were provided by both use of magnesia or sorel cement or hydrated lime reagents in the salt leaching Turkish lignites.

High ash mineral contents of Turkish lignites reduced desulfurization rate in the semi-fused salt-caustic leaching method.

Total Sulfur hold-up ratios reached only 74-67% values for Soma, Aydın, Kütahya Gediz and Denizli Çivril lignites in sorel cement and magnesia media, respectively. Those hold-up values were obtained at leaching temperatures of 300 and reduced slightly at 350-370°C.

In general, small range deterioration of coal particles in physical and chemical features over 350 °C was observed by semi-pyrolysis with lack of oxygen content in the reactor. Ash reduction ratios by caustic component and salt just occurred at low levels of 10-20%. Devolatilization ratios reached to about 25%.

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