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Original Research

Role of Inorganic Materials on Oil Agglomeration of Zonguldak Bituminous Coal Dust

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A B S T R A C T

This study investigates the oil agglomeration method which has significant importance for achieving high recovery and cost-effectiveness in utilizing energy resources. This method is particularly relevant for resources that pose environmental challenges due to their fine particle size or low quality, such as oxidized coal with large reserves. This research examines the effects of various factors, i.e. type of inorganic pretreatment material, proportion of bridging liquid, duration of agglomeration and mixing speed, on the efficiency of oil agglomeration in the beneficiation of Zonguldak coal dust, which has a low ash content of 2.23%. In the experiments, kerosene serves as the bridging liquid, and various inorganic pretreatment materials, including NaCl, $FeCl_2$, $Fe_2(SO_4)_3$ and $Al_2(SO_4)_3$, are tested. The experiments revealed that when inorganic pretreatment materials were combined with kerosene, the maximum agglomeration recovery of 81.6% was achieved at kerosene concentration of 15%, mixing speed of 800 rpm, and agglomeration time of 10 minutes. The agglomeration recovery was found to be 71.5% when using only kerosene under suitable conditions, but it varied between 78.3% and 81.6% when pretreatment materials were combined with kerosene. Among the inorganic pretreatment materials, the highest recovery was obtained with the use of 50 mg/L $Fe_2(SO_4)_3$ in combination with kerosene. Furthermore, the optimum agglomeration recovery obtained with only kerosene could be achieved in a shorter agglomeration time when inorganic pretreatment materials were used together with kerosene.

Keywords: *Bituminous coal. Bridging liquid. Coal dust. Kerosene. Oil agglomeration.*

Introduction

Today, the recovery of coal dust through cleaning and increasing the quality of coal becomes imperative along with developing technology due to both economic reasons and environmental issues. Excessive fragmentation of Zonguldak coals due to their brittle structure and applied production methods significantly increases plant losses in the fine size fractions. Of the raw coal produced in the basin, according to the enterprises, 15-30% constitutes 1 mm size coals, which can be referred to as fine coal. For this reason, the significance of fine coal enrichment increases significantly (Hacıfazlıoğlu, 2013).

The beneficiation of fine coal in coal washing plants is performed with the help of some new technology devices such as coal spirals, flotation machines, feldspar jigs, shaking tables, water cyclones, multi-gravity separators, teetered-bed separators and reflux classifiers. Heavy media beneficiation method, which is based on the density difference between coal and mineral matter, is one of the most widely used methods in coal beneficiation plants (Abbott and Miles, 1991; Aktaş, 1993; Özbayoğlu and Mamurekli, 1994; Aktaş and Woodburn, 1995; Aktaş et al., 1998; Çelik, 2002; Çelik, 2006; Yüce et al., 2009). The ash content in the floating part of the coal obtained in the beneficiation process performed with heavy media separation is equivalent to the maximum amount of mineral matter that can be removed (Flynn and Woodburn, 1987; Stockton, 1989; Aktaş, 1993; Aktaş et al., 1998; Ünal et al., 2000). On the other hand, in the heavy media beneficiation method, the recovery decreases at fine sizes, and especially the recovery of coals at sizes finer than 0.5 mm becomes quite difficult (Kemal and Arslan, 2000).

Due to the inadequacy of traditional gravity methods for fine coal beneficiation, methods like flotation, selective flotation, and oil agglomeration, which leverage the physicochemical surface properties of mineral grains, are becoming increasingly important (Cebeci and Sönmez, 2002). Flotation is a beneficiation method that is widely used in cleaning coal dust and is performed by utilizing the differences in surface properties of organic and inorganic components of coal. However, in the flotation process of oxidized coals under 0.074 mm size and coal dusts with high clay content, various technical and economic problems arise, such as high moisture in the clean coal filter cake, low recovery and poor selectivity (Aktaş, 2002).

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In the field of beneficiation studies; the flocculation method, which is used to regulate the distribution of very fine-sized coal particles (<0.15 mm) in suspension, is not economically viable (Hacıfazlıoğlu, 2013; Şahinoğlu, 2006) and the oil agglomeration method holds significant importance in terms of high recovery and low cost, particularly for the utilization of our energy resources that pose environmental challenges due to their fine particle size or for our low-quality oxidized coal resources with substantial reserves. The oil agglomeration method is a beneficiation technique capable of selective separation by exploiting differences in the surface properties of minerals associated with raw coal (-0.5 mm) or finely ground (<0.074 mm) intermediate products. Furthermore, oil agglomeration is an efficient process that can replace flotation in certain contexts, as it can reduce coal moisture in the form of oil agglomerates without the need for thermal drying, filtration or thermal dewatering. It also enables the cleaning of oxidized coals and the recovery of coal from coal washery residues (Nicol and Swanson, 1980; Guerra et al., 1986; Hazra et al., 1986; Hoşten and Uçbaş, 1989; Cebeci et al., 2002).

However, the major drawback of the oil agglomeration method is its high oil consumption (Kılınç, 2000). To mitigate this issue, recent studies have focused on optimizing the usage of oil and exploring alternative, more sustainable agglomerants that could potentially lower costs and reduce the environmental impact associated with oil agglomeration. The oil agglomeration method represents a critical advancement in coal beneficiation technologies, offering a promising approach for enhancing the utilization of coal as an energy resource. Its ability to address the specific challenges posed by fine and oxidized coals, combined with ongoing research aimed at improving its economic and environmental performance, underscores its potential as a key strategy in the sustainable management and utilization of coal reserves.

Oil agglomeration is a beneficiation method in which selective separation is performed by making use of the differences in the surface properties of coal and accompanying minerals (Kılınç, 2000). Coal consists of organic contents, inorganic compounds constituting ash and other mineral substances. Factors such as the carbon content percentage in coal, the presence of inorganic materials, and the level of carbonization influence the surface characteristics of coal, which in turn impacts its beneficiation process (Laskowski and Parfitt, 1988; Şahinoğlu, 2006; Gülsuna, 2007).

In the oil agglomeration method, fine hydrophobic coal particles are agglomerated with an oil containing hydrocarbons, and the obtained dispersion resistant agglomerates are taken from the liquid phase with a mechanical method. Hydrophilic mineral substances, on the other hand, remain in suspension in a dispersed state (Mehrotra et al., 1983; Capes and Darcovich, 1984; Capes, 1991). The separation of coal from the accompanying inorganic substances depends on the fact that both coal particles show sufficient natural hydrophobic characteristics and the oil selectively wets coal surfaces in appropriate conditions and forms spherical agglomerates by bridging particles (Capes and Germain, 1982; Slaghuis and Ferreira, 1987; Steedman and Krishnan, 1987; Petela, 1991; Shrauti and Arnold, 1995; Garcia et al., 1998; Laskowski and Yu, 2000; Düzyol, 2015).

The amount of bridging liquid strongly affects agglomerate structure, recovery of agglomerates and selectivity (Capes and Germain, 1982; Capes and Darcovich, 1984; Steedman and Krishnan, 1987; Petela, 1991), while also determining the size and appearance of agglomerates (Darcovich et al., 1989; Petela, 1991; Garcia et al., 1995; Alonso et al., 2002; Cebeci and Sönmez, 2002; Chary and Dastidar, 2010; Cebeci, 2003; Gence, 2006; Cebeci and Sönmez, 2006; Chary and Dastidar, 2013; Düzyol, 2015; Kumar et al., 2015). Agglomerants form smaller, rounder and more compact agglomerates in low concentrations, whereas larger, irregular and looser agglomerates are formed at higher concentrations (Cebeci, 2003).

Although oil agglomeration processes have been used since 1920, microscopic coal-oil-water interactions are still yet to be completely understood. This originates from the heterogeneous structure of coal, which consists of hydrophilic and hydrophobic regions (Keller and Burry, 1987; Özer et al., 2017). Coal agglomeration processes are based on the wettability difference between clean coal particles and mineral substance when coal is treated with an agglomerant or bridging agent such as light and heavy hydrocarbons or non-hydrocarbon oils. In fact, agglomeration is only possible when coal particles are hydrophobic enough to allow selective wetting with oil in place of water. Agglomeration is strongly dependent on petrography, structure, type, sequence and surface oxidation of coal in addition to agglomerant type and concentration. If the coal particle hydrophobicity is not sufficient, surface modification agents are usually added to the coal-water-oil slurry to enhance the agglomeration of clean coal particles. Besides, in some circumstances, surface active agents can be incorporated into the slurry to reduce the amount of agglomerant and oil needed in the process (Özer et al., 2017).

In the agglomeration experiments, kerosene was used as the bridging liquid because Zonguldak hard coal is bituminous coal. On the other hand, heavy oils are used in the agglomeration of coals such as sub-bituminous and lignite. Heavy oils characteristically comprise polar hydrophilic functional groups, such as nitrogen, oxygen and sulfur, and these groups promote adsorption on the relatively hydrophilic surfaces of coals to create agglomerates, whereas light oils with low density and viscosity are used in the agglomeration of bituminous coal (Capes, 1976). Light oils increase the probability of particle-oil droplets collision as they can be easily dispersed in the pulp through mixing (Capes, 1991; Ünal et al., 2000; Chary and Dastidar, 2010).

There are many studies in the literature regarding the effect of different processing factors, such as coal recovery, ash content, coal petrography and composition of agglomerates, coal particle size, pulp density, type and concentration of bridging liquid and mixing speed, on agglomeration performance in beneficiation by oil agglomeration (Capes and Germain, 1982; Mehrotra et al., 1983; Capes and Darcovich, 1984; Slaghuis and Ferreira, 1987; Steedman and Krishnan, 1987; Darcovich et al., 1989; Hoşten and Uçbaş, 1989; Capes, 1991; Petela, 1991; Yamık et al., 1994; Garcia et al., 1995; Shrauti and Arnold, 1995; Garcia et al., 1998; Kılınç, 2000; Laskowski and Yu, 2000; Ünal et al., 2000; Alonso et al., 2002; Cebeci and Sönmez, 2002; Cebeci, 2003; Cebeci and Sönmez, 2006; Gence, 2006; Ünal and Erşan, 2007; Chary and Dastidar, 2010; Kılınç Aksay et al., 2010; Uslu and Şahinoğlu, 2010; Düzyol et al., 2012; Chary and Dastidar, 2013; Düzyol, 2015; Kumar et al., 2015). The biggest disadvantage of oil agglomeration is high oil consumption. There exist a limited number of studies towards the use of surface active agents, which improve the hydrophobicity of coal and assist the oil to disperse in water, to reduce oil consumption and to obtain agglomerates with high quality in the agglomeration of dust coals (Kılınç Aksay et al., 2010).

This study involves the use of the floating part obtained from the beneficiation of hard coal from the Zonguldak-Kozlu region through heavy medium separation in oil agglomeration experiments. This research investigates the effects of variables such as bridging liquid concentration, agglomeration time, mixing speed, and the use of various inorganic pretreatment materials on recovery. This study presents significant methodological differences from similar works in the literature by emphasizing the importance of optimizing kerosene concentration and adjusting mixing speed and agglomeration time. Particularly, the maximum recovery (81.6%) achieved at 15% kerosene concentration and 800 rpm mixing speed demonstrates the critical impact of mixing speed on agglomeration efficiency. Additionally, the use of cleaned coal resulting in a homogeneous coal structure and low ash content has led to different outcomes compared to previous studies using raw coal. These differences highlight that the efficiency of the agglomeration process can vary depending on the quality of the coal used and the processing conditions.

1. Materials and Methods

1.1. Materials

In the beneficiation studies using oil agglomeration method, the samples taken from the coal mine belonging to the Turkish Hard Coal Institution, Kozlu Hard Coal Enterprise were used. Raw coal was subjected to a float-sink operation in a heavy medium, formed from $ZnCl_2$ solution, at a density of 1.3 g/cm^3. The size reduction process was applied to the floating part using a jaw crusher and a ball mill. The sample obtained from the size reduction process was sieved with a 0.5 mm sieve, and the oversize was stored, while the undersize was used in oil agglomeration experiments. The flowchart followed in sample preparation is shown in Figure 1.

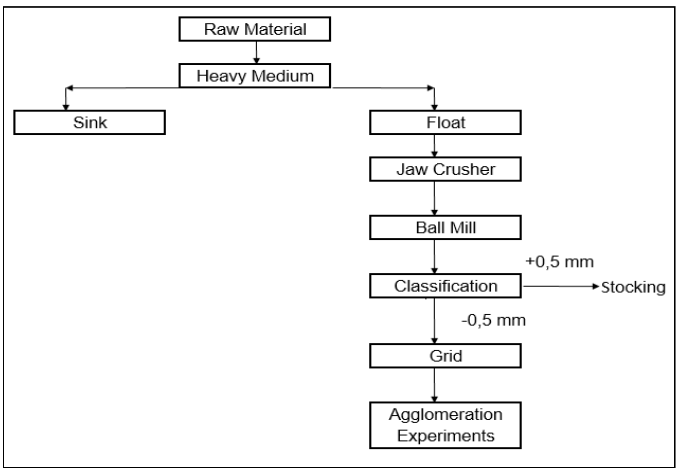

Figure 1. Sample Preparation Flowchart

Particle size analysis was performed on the coal sample prepared for agglomeration experiments. The particle size distribution curve of the coal used in the agglomeration experiments is given in Figure 2.

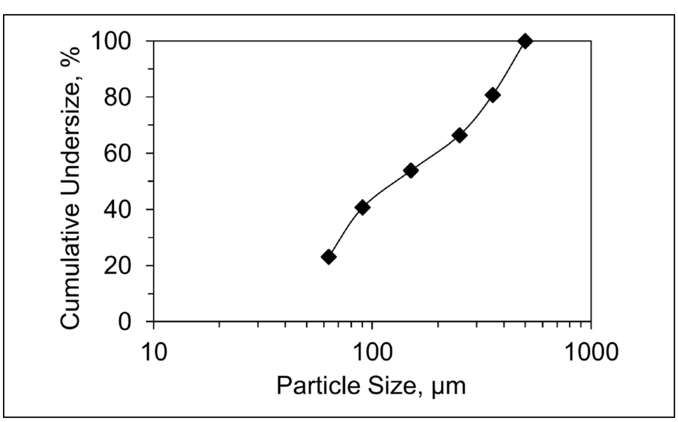

Figure 2. Particle Size Distribution of the Coal Sample

The chemical analyses of the coal used in the experimental studies were carried out using an IKA-Mag branded ash furnace for ash analysis, an IKA 4000 device for total calorific value analysis, and a LECO branded sulfur-carbon test device for sulfur analysis. The results of these analyses are given in Table 1.

Table 1. Chemical Analysis Results of the Coal Sample

Property	Value
Gross calorific value, cal/g	7790
Sulfur, %	0.30
Ash, %	2.23

In oil agglomeration experiments, kerosene (Sigma) with a density of 0.78 g/cm^3, viscosity of 1.5 cSt and 99% puritywas used as the bridging liquid. The selection of inorganic pretreatment materials, NaCl, $FeCl_2$, $Fe_2(SO_4)_3$ and $Al_2(SO_4)_3$, all from Merck, was based on a literature-based evaluation of their effects on surface properties of coal particles and how these properties influence the oil agglomeration process. The inorganic pretreatment materials were chosen to enhance the hydrophobic properties of coal particles and to facilitate more effective agglomeration during the process. NaCl and $FeCl_2$ can interfere with the agglomeration process by affecting the electrical charge distribution on coal surfaces, whereas $Fe_2(SO_4)_3$ and $Al_2(SO_4)_3$ promote the formation of hydrophobic areas on the surface, easing the interaction between oil droplets and coal particles. These inorganic pretreatment materials were selected with the aim of better understanding the mechanisms behind coal agglomeration and enhancing the effectiveness of the oil agglomeration method in coal beneficiation technologies.

1.2. Methods

Agglomeration experiments were carried out in a 50 mL beaker in which three 2 cm wide plates were present. In addition, an IKA RW-20 stirrer was used for mixing, and the mixing was ensured by a propeller with four blades having 5 cm diameter and 1 cm width, and held 1 cm above bottom of the beaker.

The experiments were conducted using 10 g coal at 10% solids ratio. A conditioning time of 5 minutes was allowed in all experiments before adding the bridging liquid. The sample obtained at the end of the experiments was sieved with a 0.710 mm sieve, and the oversize fraction was taken as the agglomerate. The agglomerates obtained were dried in an oven at 105 °C after being subjected to the cleaning process. The agglomeration recovery was determined as the ratio of the mass of agglomerate to the mass of coal fed. This recovery was employed in the evaluation of experimental results.

In the experiments in which only kerosene was used, the bridging liquid in the ratios of 2, 4, 8, 12, 15, 20, 30, and 40% were used. In the experiments carried out with only inorganic pretreatment materials, 25, 50, 250, 500, and 1000 mg/L pretreatment materials were added to the system. The agglomeration time of 10 min was given in the these experiments.

In the experiments conducted with only kerosene; when the effect of agglomeration time on the recovery was examined, the bridging liquid in the ratios of 5, 10 and 15% were incorporated to the system, and the agglomeration times of 1, 5, 10 and 15 min were applied for each ratio; in investigating the effect of mixing speed on the recovery, kerosene in the ratios of 5, 10 and 15% was added at the mixing speeds of 300, 600 and 1200 rpm, and 10 minutes of agglomeration time was allowed.

In the experiments in which inorganic pretreatment materials and kerosene were used together, a conditioning time of 5 min was given after the addition of the pretreatment materials to the system, then kerosene in the ratio of 15% was incorporated, and the agglomeration time of 10 min was applied. In these experiments, when the effect of agglomeration time on the recovery was investigated, after the addition of 5, 50 and 150 mg/L NaCl and $Fe_2(SO_4)_3$ as the pretreatment material, 5 min conditioning time was used, afterwards the bridging liquid in the ratio of 15% was incorporated to the system, and the agglomeration times of 1, 5, 10 and 15 min were given for each amount of the pretreatment materials.

In the preliminary agglomeration experiments conducted at various pH levels (3, 5, 7, 8, 10, 12) using only coal and kerosene, the effect of pH on agglomeration recovery was investigated. The highest agglomeration recovery was observed between pH 7.3 and pH 8, with recovery decreasing as the pH increased. In this study, the optimal pH value was determined to be 7.7. This represents the pH range that optimizes interaction in the agglomeration process, thereby achieving maximum recovery.

2. Results and Discussion

2.1. Agglomeration Experiments Conducted with Only Kerosene

2.1.1. Effect of Kerosene Concentration on Recovery

In the experiments in which only kerosene was used, the bridging liquid in the concentration of 2, 4, 8, 12, 15, 20, 30 and 40% were used, and the agglomeration time of 10 min was given. The recovery values obtained in the agglomeration experiments conducted with the use of kerosene only are illustrated in Figure 3.

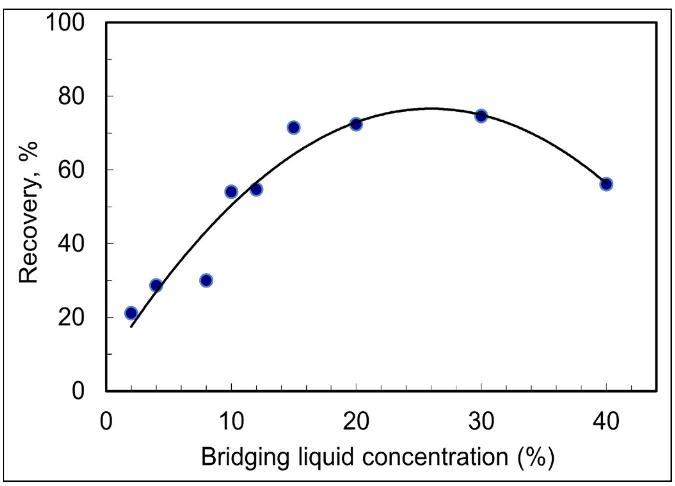

Figure 3. Recovery Variation Depending on the Bridging Liquid Concentration

The investigation of experimental data presented in Figure 3 reveals a complex interplay between the concentration of the bridging liquid and its impact on the efficiency of coal recovery via agglomeration. It was observed that recovery rates progressively increased with the concentration of bridging liquid up to 25% and beyond this point, a significant reduction in recovery was noted. This reduction at higher concentrations primarily results from spherical agglomerates transforming into less coherent, paste-like structures. These structures tend to form bulkier clusters, which diminish their structural integrity under mechanical stress (Capes and Jonasson, 1988; Hacıfazlıoğlu, 2008; Aslan and Ünal, 2011). Sub-optimal concentrations resulted in the formation of loose flocs due to insufficient oil coverage around the coal particles. This led to an increased presence of water within the agglomerates, making them prone to easy breakdown during operations, such as sieving or washing (Nicol and Swanson, 1980; Cebeci and Sönmez, 2006). Crucially, optimal agglomeration efficacy is achieved with a careful application of oil, typically maintained between 10% and 50% of the mass of coal (Hacıfazlıoğlu, 2008). Excessive application of oil was found to be detrimental to recovery, leading to the conversion of agglomerates into oil sludge and causing coal particles to form non-cohesive, paste-like flocs in an oil-rich environment (Özer et al., 2017).

Additionally, insights from experiments utilizing only kerosene have revealed that although a 25% concentration of the bridging liquid facilitated high recovery rates, the formed agglomerates exhibited irregularities and compromised durability against mechanical forces. Conversely, a 15% concentration produced denser, more spherically shaped agglomerates with superior resistance to breakage. This emphasizes the necessity of finely tuning the concentration of the bridging liquid to foster the formation of agglomerates with optimal physical characteristics and stability, highlighting the intricate balance essential in the agglomeration process to maximize coal recovery efficiency.

The findings of the study reveal that the agglomeration process achieves its highest recovery (71.5%) at a kerosene concentration of 15%, and the recovery decreases when this concentration is exceeded. Using clean coal resulted in a more homogeneous coal structure and lower ash content, differing from the results obtained with raw coal used in the study by Cebeci et al. (2002). These differences indicate that the impact of kerosene concentration on agglomeration recovery can vary depending on the quality of the coal used.

2.1.2. Effect of Agglomeration Time on Recovery

In the experiments that used only kerosene to investigate the effect of agglomeration time on recovery, bridging liquid concentrations of 5%, 10%, and 15% were added to the system. Agglomeration times of 1, 5, 10, and 15 minutes were applied for each concentration.The effect of agglomeration time on the recovery in different kerosene concentrations is shown in Figure 4.

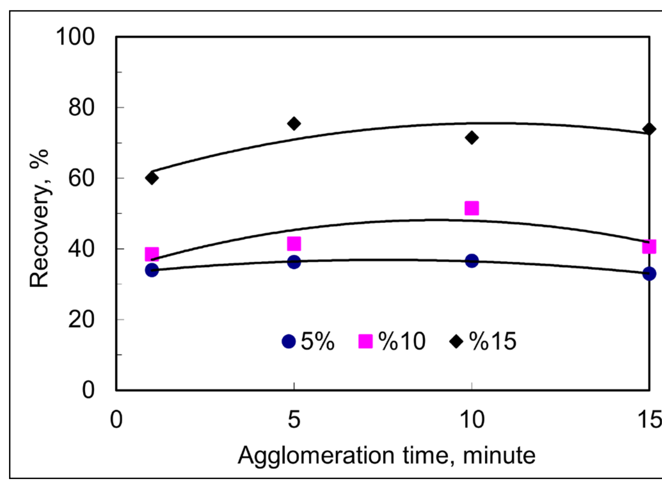

Figure 4. Effect of Agglomeration Time on Recovery at Kerosene Concentrations of 5, 10 and 15%

As shown in Figure 4, with a 15% concentration of the bridging liquid, the recovery increased from 60% to 71.5% as a result of mixing for 1 and 10 minutes, respectively. This increase in recovery was attributed to the increased likelihood of particle-oil, particle-particle and particle-microagglomerate contacts, resulting in agglomerates that were more stable, contained less water, and were more spherical.The maximum recovery (71.5%) was reached at the end of the 10 min mixing time. A slight downward trend in recovery was observed when the mixing time increased from 10 to 15 minutes. This decrease in recovery may be attributed to the agglomerates, having reached a certain size, being partially fragmented by rubbing against each other and the beaker wall. Furthermore, it was observed that the agglomerates had a looser structure, as particle-oil contacts and attachments were reduced at shorter mixing times. Similar results were reported by Osborne (1988), Uçbaş et al. (1997), Hacıfazlıoğlu (2008), Şahinoglu and Uslu (2008), Chary and Dastidar (2010) and Shukla and Venugopal (2019). Analysis of Figure 4 indicated that the optimal agglomeration time was 10 minutes.

The results of this study, compared to those of Cebeci et al. (2002), highlight significant differences in the effect of agglomeration time. Experiments conducted with clean coal achieved the highest agglomeration recovery at a kerosene concentration of 15%, with increases in this concentration negatively affecting the recovery. The maximum recovery value (71.5%) was reached within a mixing time of 10 minutes, whereas in the study of Cebeci et al. (2002) using raw coal, this duration was determined to be 15 minutes. This difference demonstrates that the efficiency of the agglomeration process varies depending on the quality of the coal used and the processing conditions. The use of clean coal, resulting in a more homogeneous and lower ash content structure, has led to different outcomes compared to previous studies that utilized raw coal.

2.1.3. Effect of Mixing Speed on Recovery

To investigate the effect of mixing speed on recovery, kerosene at different concentrations (5%, 10%, and 15%) was added to the system at mixing speeds of 300, 600, and 1200 rpm, with an agglomeration time of 10 minutes for each. The impact of mixing speed on recovery, in experiments utilizing only kerosene, is illustrated in Figure 5.

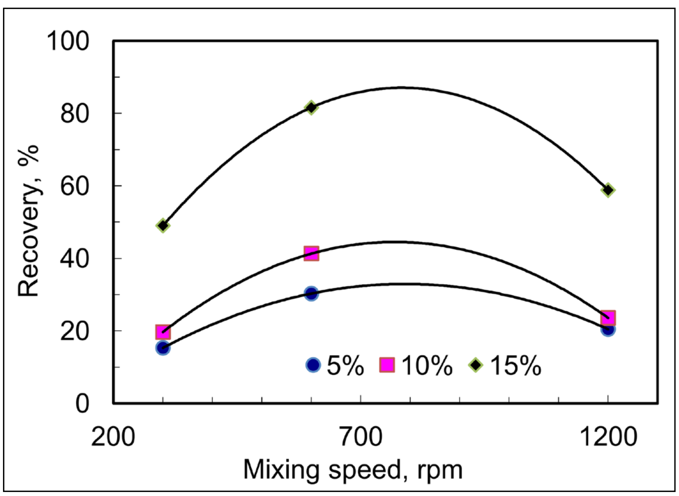

Figure 5. Recovery Variation with Mixing Speed at Kerosene Concentrations of 5, 10 and 15%

Analysis of Figure 5 shows that at a 15% bridging liquid concentration, increasing the mixing speed from 300 rpm to 800 rpm raised the recovery rate from 49% to 81.6%. However, further increasing the speed to 1200 rpm reduced the recovery to 58.8%. The diminished recovery at lower speeds is attributed to the inadequate dispersion of oil in the pulp and reduced collisions among oil-coated particles (Bhattacharyya et al., 1977; Coleman et al., 1995; Aslan and Ünal, 2011). As the mixing speed increases, the bridging liquid more effectively coats particle surfaces, enhancing the likelihood of collisions between oil-coated particles and thus improving recovery. The decline in recovery beyond a critical mixing speed is due to high shear and turbulence (Yu, 1998), uneven distribution of oil droplets (Capes and Germain, 1982; Coleman et al., 1995), and collisions of coal-oil agglomerates with each other and the beaker walls at high speeds (Cebeci and Sönmez, 2002; Kumar et al., 2015). The literature indicated that agglomeration recovery increases with mixing speed up to a critical point, beyond which it decreases (Cebeci and Sönmez, 2006; Gence, 2006; Şahinoğlu and Uslu, 2008; Aslan and Ünal, 2011; Duzyol, 2015; Özer et al., 2017). Upon reviewing Figure 5, the optimal mixing speed was identified as 800 rpm.

2.2. Agglomeration Experiments Conducted with Only Inorganic Pretreatment Materials

The agglomeration recovery values obtained from agglomeration experiments conducted using only inorganic pretreatment materials indicated that, in the absence of kerosene as a bridging liquid, the effectiveness of all tested inorganic pretreatment materials in coal agglomeration was negligible, with agglomeration efficiencies being less than 1%. This outcome underscores the critical role of bridging liquids in facilitating agglomeration, as inorganic pretreatment materials alone, without the presence of a bridging agent, fail to achieve significant dispersion on particle surfaces or to exhibit bridging properties.

2.3. Agglomeration Experiments Conducted with Inorganic Pretreatment Materials and Kerosene

In the experiments in which inorganic pretreatment materials and kerosene were used together, after the addition of the pretreatment material, a conditioning time of 5 min was allowed, then kerosene in the concentration of 15% was added to the system and 10 min agglomeration time was given.

2.3.1. Agglomeration Experiments Conducted with $Al_2(SO_4)_3$ and Kerosene

The recovery values obtained as a result of the oil agglomeration experiments conducted with $Al_2(SO_4)_3$ were given Figure 6. An increase in $Al_2(SO_4)_3$ concentration to 50 mg/L led to an enhanced recovery, after which a slight decrease was observed. The initial recovery improvement at lower concentrations can be attributed to electrostatic attractions facilitating better cohesion among coal particles and their interaction with oil droplets. In contrast, at higher concentrations, electrostatic repulsion may have impeded effective agglomeration due to the partial surface coating of coal particles with $Al(OH)_3$, thereby preventing direct contact between oil droplets and coal surfaces.

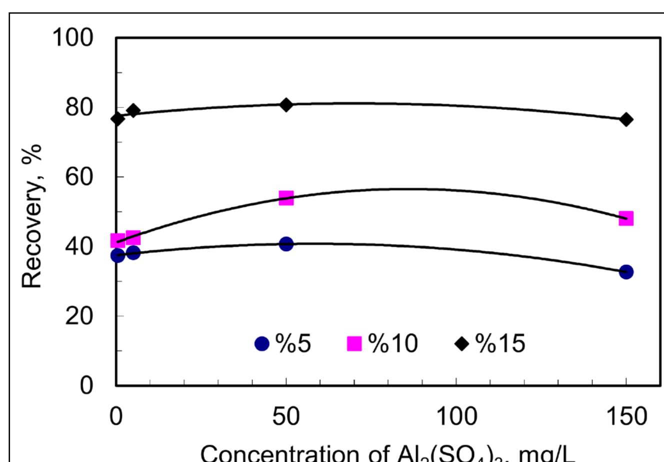

Figure 6. Recovery Variation with respect to $Al_2(SO_4)_3$ Concentration (Kerosene concentration: 5, 10 and 15%; pH=7.5)

These observations align with previous research indicating that $Al_2(SO_4)_3$, among other salts, influences the electrokinetic properties of coal and oil droplets, affecting their interactions and, ultimately, the efficiency of the agglomeration process. Notably, this study observed a 12.9% increase in recovery with 50 mg/L $Al_2(SO_4)_3$ at a 15% bridging liquid concentration, highlighting the nuanced role of inorganic pretreatment materials in optimizing the agglomeration process.

Furthermore, salts such as $Al_2(SO_4)_3$, $CaCl_2$, NaCl, and $FeSO_4$ have been shown to impact the zeta potential of oil droplets (Wen and Sun, 1981; Gurses et al., 1997). The ions $Al(OH)_2^+$ and $Al(OH)_4^-$ are found to affect the electrokinetic potential of coal particles and oil droplets (Özbayoglu, 1980; Cebeci et al., 2002), contributing directly to the agglomeration process's efficiency. This study underscores the importance and potential effects of using inorganic pretreatment materials in the agglomeration process, providing detailed insights into their contribution to the field of coal beneficiation technologies.

In this study, the use of clean coal resulted in an increase in agglomeration recovery with the increase of $Al_2(SO_4)_3$ concentration up to 50 mg/L, followed by a subsequent decrease. This indicates that electrostatic attractions facilitated better cohesion among coal particles and their interaction with oil droplets. In contrast, the study by Cebeci et al. (2002), which used raw coal, reported a decrease in combustible recovery with the increase of $Al_2(SO_4)_3$ concentration up to 75 mg/L, but then observed a partial increase. This difference highlights the effects of the type of coal used (clean coal vs. raw coal) on the agglomeration process and the role of inorganic pretreatment materials. Both studies underscore the complexity of $Al_2(SO_4)_3$'s impact on the agglomeration process and the significance of the coal type used.

2.3.2. *Agglomeration Experiments Conducted with $Fe_2(SO_4)_3$ and Kerosene*

The recovery values obtained as a result of the oil agglomeration experiments in which $Fe_2(SO_4)_3$ was used as the inorganic prematerial are illustrated in Figure 7. As seen in Figure 7, similar to the trend observed in $Al_2(SO_4)_3$ experiments, an increase in recovery was noted with the concentration of $Fe_2(SO_4)_3$ up to 50 mg/L, followed by a slight decrease. The increase in recovery at low concentrations can be attributed to the enhanced hydrophobicity of coal particles due to the adsorption of Fe^{3+} ions on their surface. Conversely, the decrease in recovery at high concentrations may be linked to the obstruction of oil droplets contact with coal particles, presumably because the $Fe(OH)_3$ compound, believed to be present in the medium, partially coats the coal particle surface. Similar findings have been reported in studies by Butler (1964), Özbayoğlu (1987) and Cebeci et al. (2002). Evaluating the results from agglomeration experiments with $Fe_2(SO_4)_3$, it was observed that 50 mg/L of $Fe_2(SO_4)_3$ increased the recovery by 14.1% at the bridging liquid concentration of 15%.

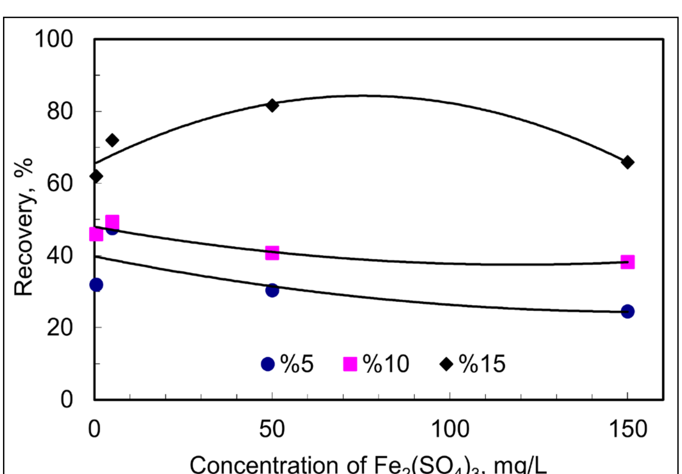

Figure 7. Recovery Variation with respect to $Fe_2(SO_4)_3$ Concentration (Kerosene concentration: 5, 10 and 15%; pH=7.8)

In this study, the increase in recovery at low concentrations has been linked to the adsorption of Fe^{3+} ions on the surface of coal particles. The adsorption of Fe^{3+} ions enhances the hydrophobicity of coal surfaces by forming complex bonds with oxygen-containing functional groups, such as carboxyl (-COOH) and hydroxyl (-OH), present on the coal surface. These complex bonds reduce the interaction potential of the coal surface with water molecules, thereby giving the surface a more hydrophobic character. This, in turn, facilitates easier coalescence of coal particles with oil droplets during the oil agglomeration process, leading to an increase in agglomeration efficiency. Research conducted by Arbiter (1985), Wen and Sun (1981), and Langmuir (1969) has shown that the adsorption of Fe^{3+} ions significantly enhances the hydrophobicity of coal surfaces, thereby improving the efficiency of oil agglomeration.

2.3.3. *Agglomeration Experiments Conducted with $FeCl_2$ and Kerosene*

The recovery results obtained from the oil agglomeration experiments conducted with $FeCl_2$ are shown in Figure 8. As seen in Figure 8, the recovery slightly decreased with an increase in the concentration of $FeCl_2$. This decrease in recovery was attributed to the fact that Fe^{2+} and $Fe(OH)^+$ cations, which are believed to be present in the medium, render the surfaces of coal particles and oil droplets more hydrophilic by altering their surface charge (Frederick, 1964; Langmuir, 1969; Wagman, 1969; Fuerstenau et al., 1983; Gutierrez-Rodriguez and Aplan, 1984; Arbiter, 1985; Özbayoğlu, 1987; Laskowski and Parfitt, 1988). Additionally, the $Fe(OH)_2$ compound, also presumed to exist in the medium albeit in smaller quantities, prevents coal particles from contacting the bridging liquid (Butler, 1964; Fuerstenau and Palmer, 1976; Özbayoglu, 1980; Wen and Sun, 1981; Gurses et al., 1997). The results from the experiments showed that 50 mg/L $FeCl_2$ increased the recovery by 11.1% at a bridging liquid concentration of 15%.

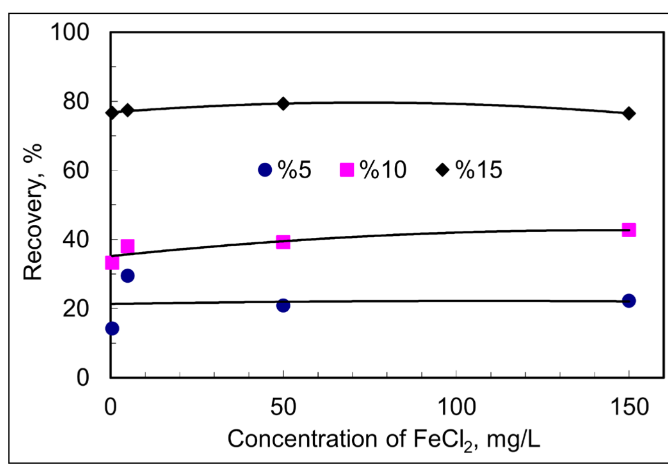

Figure 8. Recovery Variation with respect to $FeCl_2$ Concentration (Kerosene concentration: 5, 10 and 15%; pH=7.3)

2.3.4. *Agglomeration Experiments Conducted with NaCl and Kerosene*

The recovery values obtained from the oil agglomeration experiments conducted with NaCl are presented in Figure 9. As seen in Figure 9, the evaluation of experimental results showed that adding 50 mg/L NaCl increased recovery by 9.5% at a 15% bridging liquid concentration. The increase in recovery with rising sodium chloride concentration in oil agglomeration is attributed to the reduced thickness of the electrical double layer between coal particles and oil droplets (Özbayoglu, 1980; Wen and Sun, 1981; Cebeci et al., 2002). Fan et al. (1988) explored the impact of NaCl on the oil agglomeration recovery of coals with varying hydrophobicity levels. They discovered that as NaCl concentration increased, agglomeration recovery improved in highly hydrophobic coals but decreased in less hydrophobic ones. Yang et al. (1987) demonstrated that recovery slightly increases in oil agglomeration as NaCl concentration rises, attributing this improvement to the compression of the electrical double layer between coal particles and oil droplets. The analysis of experimental data confirmed that 50 mg/L NaCl enhanced recovery by 9.5% at a 15% bridging liquid concentration.

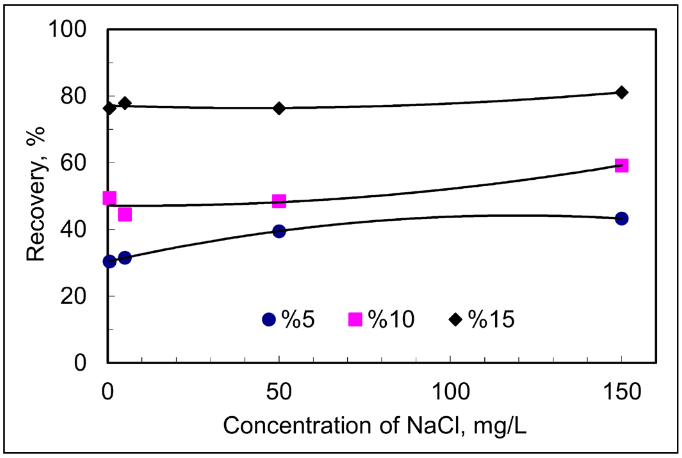

Figure 9. Recovery Variation with respect to NaCl Concentration (Kerosene concentration: 5, 10 and 15%; pH=7.7)

In this study, the results showed that adding NaCl to a mixture with a 15% kerosene concentration and using clean coal led to a 9.5% increase in recovery. These findings align with those of Cebeci et al. (2002), who used raw coal, highlighting NaCl's influence on the agglomeration process. However, the substitution of raw coal with cleaned coal altered the process's efficiency, yielding a more uniform coal structure with lower ash content and, consequently, different outcomes.

2.4. Comparison of the Results of Experiments Conducted with Different Inorganic Pretreatment Materials at Different Agglomeration Times

In the experiments where the effect of agglomeration time on recovery was investigated using different inorganic pretreatment materials and kerosene, after the addition of 50 mg/L of NaCl, $FeCI_2$, $Al_2(SO_4)_3$ and $Fe_2(SO_4)_3$ as pretreatment materials to the suspension, a conditioning time of 5 minutes was allowed. Then, the bridging liquid at a concentration of 15% was added, and agglomeration times 1, 5, 10 and 15 minutes were applied for each concentration. To determine the effect of mixing time on agglomeration, four different mixing times were tested at the mixing speed of 800 rpm. The results obtained from these experiments are shown in Figure 10.

As can be seen from Figure 10, the recovery was found to be low when the agglomeration time was 1 minute. The particle-oil contacts and particle-oil attachments were low at short mixing times (Bhattacharyya et al., 1977; Osborne, 1988; Coleman et al., 1995; Hacıfazlıoğlu, 2008; Şahinoğlu and Uslu, 2008; Aslan and Ünal, 2011; Shukla and Venugopal, 2019). When the agglomeration time was increased from 1 to 5 min, recovery increased due to increased contact of oil coated particles (Capes and Germain, 1982; Kumar et al., 2015; Özer et al., 2017). From 10 to 15 min, a slight downward trend was observed in recovery. This decrease may be due to the fact that, at longer mixing times, agglomerates reaching a certain size were partially fragmented by rubbing against each other and the wall of the beaker (Coleman et al., 1995; Yu, 1998; Cebeci and Sönmez, 2002; Özer et al., 2017). In experiments conducted with only kerosene, the maximum recovery was 71.5% when the agglomeration time was 10 min. In experiments where different pretreatment materials were used (NaCl, $FeCI_2$, $Al_2(SO_4)_3$ and $Fe_2(SO_4)_3$) with kerosene, the recovery values were found to be 9.5%, 11.2%, %12.9% and 14.1%, respectively, when the agglomeration time was 5 minutes.

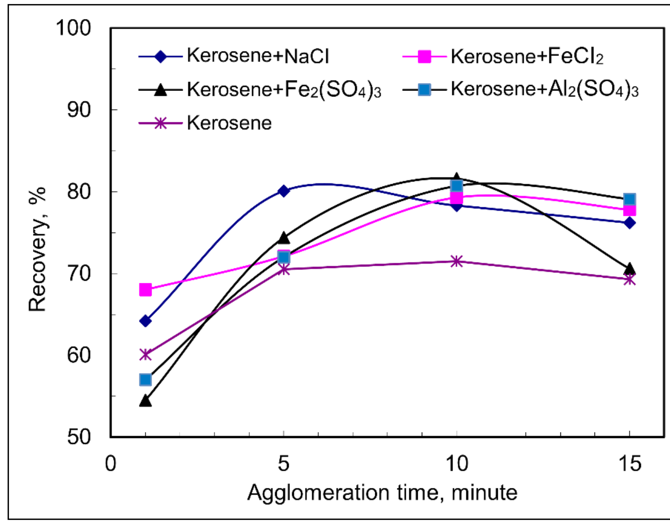

Figure 10. Recovery Variation with respect to Agglomeration Time (Pretreatment material: 50 mg/L; Kerosene: 15%)

3. Conclusions

The results of the agglomeration experiments conducted only with kerosene indicated that recovery increased up to a bridging liquid concentration of 25%, and then decreased beyond this concentration. Diminished recovery at lower bridging liquid concentrations resulted from inadequate coverage of the coal surface, whereas the decline at higher concentrations occurred because the agglomerates formed had lower resistance to mechanical forces.

In the experiments where the effect of agglomeration time on recovery was investigated using only kerosene, it was determined that the optimal time for agglomeration of hard coal was 10 minutes.

Coal could not be agglomerated using only pretreatment materials without a bridging liquid.

Using inorganic pretreatment materials led to specific increases in recovery rates, ranging from 9% to 15%, at a 15% bridging liquid concentration. This variation in recovery rates, is dependent on the specific type of pretreatment material used. Notably, the employment of $Fe_2(SO_4)_3$ and $Al_2(SO_4)_3$ resulted in significant recovery improvements, with increases of 14.1% and 12.9%, respectively. These findings imply that such materials potentially augment the hydrophobic qualities of coal particles, thereby promoting agglomeration and consequently elevating recovery rates. Conversely, the utilization of $FeCl_2$ and NaCl was associated with more modest recovery increments, 11.2% and 9.5% respectively, suggesting that these substances may restrict agglomeration efficiency. This limitation appears to stem from modifications in surface charges induced by these materials, which in turn adversely impact the agglomeration process.

Recovery levels achieved in 10 minutes with only kerosene were reached in just 5 minutes when pretreatment materials were added. This indicates that inorganic pretreatment materials can shorten the agglomeration time, thereby increasing process efficiency.

Experiments showed that maximum recovery occurred at a mixing speed of 800 rpm. This finding demonstrates the significance of mixing speed as a parameter in the agglomeration process and suggests that selecting an optimal speed can maximize recovery efficiency.

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