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An Overview of the Development of Chemical Desulfurization Technology for Organic Sulfur Coal

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ABSTRACT

Coal is a highly valuable energy source. It is used to generate electricity in power plants and is one of the most cost-effective fuels, becoming less expensive than natural gas and crude oil. Sulfur oxide is produced when high-sulfur coal is burned and reacts with water and oxygen to cause acid rain and pollution. Various chemical coal desulfurization techniques currently remove only inorganic sulfur, such as pyrite and sulfur ether, whereas organic sulfur, such as thiophene, is harder to remove. Organic sulfur is covalently bound to coal's macromolecular backbone, and the C-S bond must be broken to remove the sulfur efficiently. Coal's complex molecular structure and properties would be lost throughout this process. Chemical desulfurization was the most effective method of removing inorganic and organic sulfur. This method overcomes the limitations of conventional physical desulfurization, which primarily removes inorganic sulfur and can be lengthy in some cases. This article covered the development of chemical desulfurization technologies for organic sulfur coal, including acid and alkaline leaching, peroxyacetic acid oxidation, electrochemical or electrolysis desulfurization, microwave irradiation, and ultrasonic-assisted extraction.

Keywords: *Desulfurization, acid-alkali reagents, peroxyacetic acid, microwave irradiation, ultrasonic-assisted extraction*

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INTRODUCTION

Coal is the cheapest type of fossil fuel [1] and a reliable energy source due to its worldwide abundance and stable price [2-3]. It generates power in Malaysia, accounting for around 90% of the total demand. Industry uses fewer than 2,000 ktoe regularly. In 2010, the demand for power plant energy reached 17,101 ktoe [4]. Combusting this abundant fuel causes pollution, primarily sulfur dioxide [5].

Sulfur levels in coal range from 0.5 to 11%, with coal containing more than 3% sulfur classified as high sulfur [6]. In coal, sulfur is present mainly in pyritic, sulfate, and organic forms [7-10]. Organic sulfur compounds include mercaptans (-SH), thiophene, disulfide (R-S-S-R), and thioether (R-S-R) [11-13], whereas inorganic sulfur compounds include pyrite and sulfate [14]. On the other hand, sulfate sulfur concentration is comparatively low [12].

During combustion, organic sulfur compounds emit significant sulfur oxide and hydrogen sulfide [15-16], whereas pyritic and sulfate sulfur break down to produce inorganic oxides and SO_x. This significant amount of high-Gross Calorific Value (GCV) coal cannot be used in thermal power plants unless the sulfur content and SO_x emissions are reduced [8]. The simplest physical method for extracting inorganic sulfur from coal has little effect on organic sulfur. Because of the complexity of the coal molecule, chemical desulfurization is required [13, 17]. Organic sulfur can only be removed by destroying chemical bonds [18].

Desulfurization of coal is critical for generating clean, high-quality coal while reducing air pollution generated during combustion. For pre-desulfurization, physical, physicochemical, chemical, and biological desulfurization methods have all been developed [19-21]. Chemical desulfurization is one of the most efficient methods of eliminating organic sulfur from coal. Indeed, efficient desulfurization technologies have been developed by combining several processes [3, 17]. This method is overcome by traditional physical and biological desulfurization, which is time-consuming and ineffective in removing organic sulfur [22-23]. This study aims to compare five desulfurization methods for organic sulfur. The progress of chemical coal desulfurization has been discussed. Among these are acid and alkaline leaching, peroxyacetic acid oxidation of coal, electrochemical or electrolytic desulfurization, microwave irradiation and ultrasonic-assisted extraction.

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Acid and alkaline leaching

Coal leaching using acids and alkalis is the most successful technique for extracting pyrite and organic sulfur [23], as chemical solvents quickly permeate into the coal matrix and dissolve the minerals [24]. Traditional acid or alkali immersion methods may remove different kinds of sulfur from coal. These techniques damage the C-C structure and coal adhesion, lowering the calorific value of the coal. The corrosive nature of different acids and alkalis makes wastewater discharge harmful to the environment. To minimize acid and alkali damage to the coal structure, desulfurization will be done with an organic solvent and acid or alkali. Because acid or alkali may remove organic sulfur from organic solvents to make an inorganic salt, mixing organic solvents with acid or alkali has a more significant desulfurization impact [3]. According to Behera et al. [25], only such alkaline treatments remove organic sulfur coal. The percentage reduction, however, was small and only involved treatments with a combination of acid and alkaline solutions.

During the desulfurization of high-sulfur Indian coal, the influence of potassium hydroxide (KOH) and reaction time on coal characteristics was studied. A KOH concentration of 5-20% and 6-24 hours leaching duration were found to remove 45-49% pyritic sulfur, 24-33% sulfate sulfur, and 21-31% organic sulfur. Because the KOH concentration disrupted the C-S bond, sulfur elimination increased as contact duration increased. It was also linked to sulfate solubilization, pyritic conversion, and the conversion of specific functional groups to soluble salts, such as thiols and disulfide [8].

Several studies [25-27] have attempted to target acidic coal leaching for coal desulfurization. The coal was treated with a 15% hydrogen peroxide (H_2O_2) solution at 25 °C. The findings showed that this leaching action eliminated over 76% pyritic sulfur, 70% sulfate sulfur, 5% organic sulfur, and 14% ash. Again, leaching the remaining H_2O_2-treated coal with 0.1 N sulfuric acid (H_2SO_4) indicated complete removal of inorganic sulfur, over 26% organic sulfur removal, and a 43% reduction in ash content. The significant quantity of sulfur removed was identified due to the usage of sulfuric acid, which acts as a catalyst for the interaction between the oxygen and pyrite particles [25]. Alkali and acid leaching were shown to be less effective than a combination of the two [28]. Combinations of acid and alkali have a higher effect on desulfurization because alkali, at high temperatures, may damage the organic structure of coal and interacts with acid to create soluble salts of sulfur in coal. On the other hand, acid and alkali treatments damage coal's combustible organic macromolecular structure, lowering its calorific value after desulfurization [3]. Combining ultrasonic treatment with chemical leaching will be covered in the next section.

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Oxidation of coal by peroxyacetic acid

Oxidation is the most widely used process for coal desulfurization [17, 29-32] due to its ability to remove the pyritic sulfur [33] and the macromolecular network [34]. Various factors influence coal oxidation, including reagent concentration, processing temperature, time, pressure, coal type, and particle size. Common oxidizing agents used on coal include hydrogen peroxide, potassium permanganate, chlorine, and other chemical oxidants. While potassium permanganate and chlorine are both excellent desulfurizes, they have been found to harm the microstructures and properties of coal [5]. The research discovered that combining chemicals for coal pretreatment gave better results than using only one reagent [3, 35-36]. Under microwave heating, the combination of sodium hydroxide (NaOH) and H_2O_2 was an effective alkali desulfurizing agent with a total sulfur reduction of 55%. At the same time, the acidic reagent nitric acid, HNO_3-H_2O_2, had the highest performance, with a total sulfur reduction of 100% [35].

Ishak et al. [5] discovered that mixing 6-30% peroxyacetic acid and acetic acid at 28, 50, and 104 °C may reduce pyrite and organic sulfur forms. PAA is a weaker acid and a well-known desulfurization oxidant [37] that was insufficiently effective in organic sulfur [13]. Although microwave irradiation effectively removed mercaptan, pyrite, and thioether, heterocyclic sulfur in coal, such as the thiophene ring, remained intact due to structural stability [13]. The research discovered that combining microwave irradiation and PAA treatment changed the physical properties of coal [17] and had the potential to destroy the coal's original qualities due to its strong oxidizing properties [17, 38]. To extract thiophene from coal, the C-S bonds must be broken. Until now, the removal process of sulfur-containing bonds for thiophene in coal has been difficult to understand [13]. PAA is expected to generate hydroxyl cations as a moderate oxidizing agent. Hydroxyl cations, which are more nucleophilic than carbon atoms, react with sulfur atoms [32].

Jorjani et al. [29] stated that when coal was oxidized with PAA and leached for 40 minutes at 45 °C, organic-sulfur removal improved by 2.5%. However, the elimination of pyritic sulfur reduced over time. The PAA solution prevented sodium butoxide penetration into the coal mass, resulting in poor organic and pyritic sulfur removal. They observed pre-bleaching the PAA-oxidized sample with sodium butoxide enhanced organic and inorganic sulfur removal. The remaining PAA in the coal pores may be eliminated by heating the sample to 100 °C, and the access of the sodium butoxide solution to the sulfur atoms can be enhanced to 34% for organic sulfur and 67% for pyritic sulfur. This indicates that the organic sulfur in PAA-oxidized coal was converted to sulfones and sulfoxide, easily removed by heating with sodium butoxide [29]. The oxidizing agent's ability to remove organic sulfur was typically low. Although the PAA demonstrated a complete desulfurization impact of organic sulfur from coal, it could not selectively break the C-S bond in the thiophene ring, potentially destroying its natural character [13].

Current research has focused on using external energy fields such as ultrasonic or microwave in conjunction with PAA. According to Tang et al. [39], sulfur reduction from coal using

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microwave and chemical additions has various advantages. Both inorganic and organic sulfur in coal may be successfully eliminated by microwave heating and chemical additions because of the synergistic impact of the two processes. The microwave combined with PAA may remove around 50% of the organic sulfur in coal. Furthermore, due to the selective heating effect of microwaves, the coal matrix can remain at a low temperature. At the same time, the sulfur-containing group was rapidly heated to the reaction temperature, reducing the damage to the original coal qualities during desulfurization [39]. The following two subtopics provide more details on microwave irradiation and ultrasonic-assisted extraction.

Electrochemical or Electrolysis Desulfurization

Electrochemical desulfurization has gained popularity due to its low pressure and temperature requirements, which make it more cost-effective and energy-efficient [40-42]. The use of electrochemical desulfurization technology to remove sulfur from fossil fuels by electrochemical oxidation or reduction of sulfur molecules has been studied [42]. According to Hong-Xi et al. [43], the technique effectively removes pyritic and organic sulfur and ash [43]. Electrochemical desulfurization can be used to convert organic and inorganic sulfur to sulfates. However, whereas inorganic sulfur was quickly removed from coal through electrolysis, the removal efficiency of organic sulfur could have been higher due to the complicated structure of organic sulfur [41].

The electrochemistry method successfully removed pyrite, sulfate, and organic sulfur from Xiaoyi coal using NaOH as the electrolyte. Total sulfur removal was up to 56%. However, electrolysis removes sulfur from coal rather than soaking, which only removes trace amounts of inorganic sulfur sulfates. Alkaline electrolysis creates Fe^{3+} from pyrites, causing coal to precipitate and become unfilterable. This increased Fe content and enrichment. To summarize, pyrite coal oxidizes to form soluble sulfates easily removed by washing and filtration. It will reduce sulfur. During electrolysis, an oxidation-reduction process occurred, replacing S with O. A higher mass fraction of C is obtained by replacing S with O. Infrared spectroscopy was utilized to identify the coal sample to validate the electrolysis removal of organic sulfur coal. Electrolysis weakens the carbonyl group's absorption peak by breaking the carbon-oxygen bond. A lower carbon-oxygen ratio was achieved through hydrogenation. This means that the electrolysis procedure did not influence the molecular structure of coal. The absorption peak for the aromatic disulfide group was reduced after electrochemical desulfurization, indicating that organic sulfur was removed from the coal. However, the mechanism of electrochemical coal desulfurization is still unclear [44].

Researchers are increasingly utilizing sonochemistry to improve electrochemistry. It is a collaborative technique using ultrasonic and electrochemical technologies. This desulfurization method is uncommon. In high-sulfur coal, ultrasound can desulfurize flotation and enhance pyrite separation. Coal may be desulfurized by ultrasonically using electricity [43].

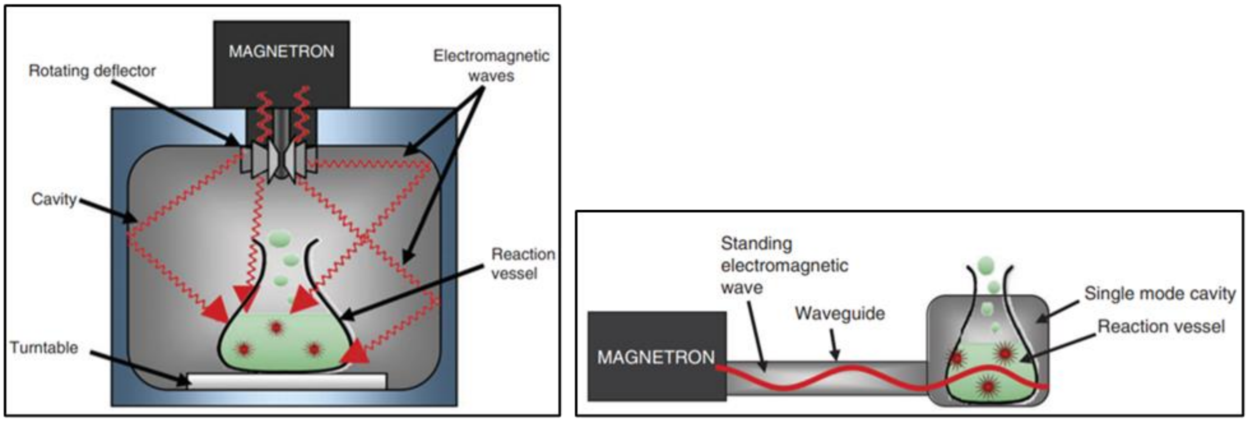
Microwave Irradiation

Microwave is an efficient heat treatment technique [45]. Coal treatment using microwaves offers excellent efficiency because they offer high penetrability, low energy usage and time savings. Furthermore, microwave treatment technology produces no pollutants during the heating process, is simple to build and install, and has easy operation suitable for automatic control [46-48]. Microwave extraction is a separation method that employs electromagnetic waves with frequencies ranging from 300 MHz to 300 GHz [39, 49-51]. Microwave ovens for the home and industrial use typically operate at a frequency of 2.45 GHz, corresponding to a wavelength of 12.2 cm and an energy of 1.02 105 eV [39]. However, microwave energy is insufficient to break chemical bonds, and the optimal activation frequencies differ for various sulfur atom types [9].

Many reported microwave chemistry experiments are carried out at 2450 MHz wavelength 12.24 cm since this frequency is approved globally and is utilized in commercially available microwave chemistry equipment. One explanation is that microwave energy absorption by liquid water is most excellent at this frequency [50-51]. When microwave radiation is applied to different materials, the properties of the materials change. Dielectrics are materials that absorb these waves better than others, such as coal [39, 52-53]. Other materials appear to be microwave radiation transparent, while others appear to reflect the radiation [39, 52]. The quantity of organic sulfur removed is determined by the microwave irradiation responses of different sulfur components, depending on their dielectric properties [54].

The most common are multimode applicators which range from home ovens to big manufacturing dryers. They are frequently formed of a "cavity," a closed metal box with at least two dimensions longer than half the wavelength. As microwaves reflect off the hollow walls, several resonance modes occur. Wave interference occurs due to reflections. Because of this, whether the load is stirred or spun, the microwave field exposed to the heated load material is very non-uniform. These applicators can be mass-produced in large quantities [52]. The multimode microwave applicator is shown in Figure 1(a). Monomode applicators, on the other hand, provide a steadier single-standing wave. The irradiated material is generally near a field maximum. Monomode applicators have limited substance capacity. Therefore, it is often utilized in as little as 200 ml. On the contrary, single-mode cavities produce greater electromagnetic field densities inside the cavity, resulting in quicker heating rates. While the electromagnetic field pattern is clearly defined in an empty single-mode cavity, adding heated material changes the pattern significantly [52]. The monomode microwave applicator is shown in Figure 1(b).

Microwave (MW) radiation is absorbed more efficiently by sulfur-containing compounds than by sulfur-free molecules with equivalent structures. Furthermore, sulfur-containing compounds' oxidation products have a greater rate of loss. Frequency has a considerable impact on the dielectric loss factor and dissipation factor of sulfur-containing compounds. Their different peaks at different frequencies suggest that specific microwave-irradiation frequencies can heat organic sulfur functional groups in coal selectively [9, 55]. The combination of PAA and MW irradiation is believed to remove a significant amount of sulfur [56]. Coal sulfide is oxidized to sulfoxide and sulfone via microwave and PAA desulfurization. Increased microwave absorption efficiency and decreased C–S bond dissociation energies and times reduce the barrier to desulfurization. These processes help to break down sulfur-containing bonds and establish desulfurization reactions. Microwave assisted-PAA desulfurizes organic sulfur at a rate of 49% [29, 55-57].



(a) (b)

Figure 1: (a) Multimode microwave applicator; (b) Monomode microwave applicator [52]

The microwave-assisted technique separates the components from the matrix using electromagnetic fields [47]. According to Ma et al. [9], the water trapped within coal pores quickly absorbs microwave radiation. Moisture and minerals such as pyrite may increase the microwave absorption of coal, preventing significant changes in its characteristics. Microwave irradiation has significantly increased the rate of several desulfurization processes. However, there are limited studies on organic compound desulfurization [9].

According to Tang et al. [58], oxidation additives are most likely to target sulfur atoms of sulfur ether in coal. In the presence of peroxyacetic acid and microwave thermal irradiation, dibenzyl sulfide (DS) and diphenyl disulfide (DDS) was desulfurized to diphenyl sulfoxide or diphenyl sulfone, respectively. Oxidizing DS formed dibenzyl sulfone to dibenzyl sulfoxide. However, certain C-S bonds in DS and its oxidation product were broken during oxidation, resulting in sulfur-containing free radicals. The broken C-S bonds resulted in water-soluble sulfonate, sulfonic ester, and sulfate. The reaction path of diphenyl sulfide and PAA is represented in Figure 2. Their sulfur atoms were oxidized to sulfones, sulfoxides, or sulfonic acids, reducing the energy of the C-S bonds [17, 58].

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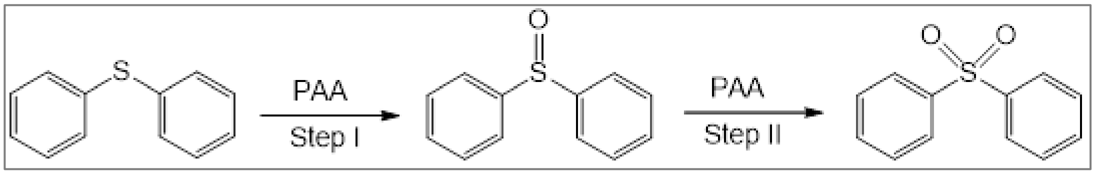

Figure 2: The reaction path of diphenyl sulfide and PAA [58]

Desulfurization of coal utilizing bromine water and microwave irradiation in Shanxi province revealed that the best desulfurization outcome occurred after five minutes of irradiation. On the other side, desulfurization decreases with increasing irradiation time, indicating that bromine water cannot penetrate the coal's pore structure. Coal and bromine water react to form a barrier that prevents bromine water from diffusing into the coal matrix. One might conclude that as the duration of the irradiation increased, the concentration of bromine water decreased due to the reaction limit for bromine water to penetrate the coal pore structure. Furthermore, the study found that microwaves coupled with bromine water had a more significant effect than bromine water alone [47].

The treatment of coal with peroxyacetic acid (PAA) led to the complete desulfurization of aliphatic organic sulfur [59-60], including thiols and thioether [13]. Pyrite, mercaptan, and thioether sulfur were quickly removed by PAA with microwave irradiation [13], while heterocyclic sulfur, such as the thiophene ring in coal, was unaffected due to structural stability [13, 17]. The C-S bonds in thiophene must be broken to remove thiophene from coal efficiently. Until recently, the method for removing thiophene sulfur from coal has remained unclear, especially the mechanism for breaking down sulfur-containing bonds in thiophene at the molecular level [13]. Combining microwave irradiation with PAA treatment changed the physical features of coal [17] and could destroy its original characteristics due to its high oxidizing capabilities [17, 38, 61].

According to Yang et. [38], the desulfurization of Gedui high sulfur coal indicated a reduction of 30% sulfate sulfur, 33.3% pyritic sulfur, and 20.1% organic sulfur following microwave irradiation with HAc-H_2O_2. Microwave treatment offers non-toxic, ecologically friendly chemicals compared to traditional heating processes. The temperature was kept constant at 105 °C using the microwave technique, while the desulfurization efficiency using conventional heating was lower. Furthermore, both organic and inorganic sulfur can be reduced concurrently. The heat from microwave irradiation helps reduce sulfur in coal.

Ultrasonic assisted extraction

Ultrasonic assistance technology has gained popularity for improving coal cleaning efficiency by combining robust streaming and cavitation effects with reduced treatment time, energy

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consumption, and reagent usage. An acoustic environment causes shock waves when bubbles collapse. The boiling point of a liquid decreases with decreasing impact force. If the pressure is reduced enough, the liquid will boil spontaneously. The localized pressure reduction causes the production of tiny vapor bubbles. Cavitation occurs when these bubbles collapse during the compression cycle [62].

Acoustic cavitation occurs between 20 and 40 kHz, whereas acoustic streaming occurs between 400 kHz and 1 MHz [63-64]. During ultrasonication, the cavitation effect increases the quantity of coal ash medium, which may affect its properties due to coal's temperature sensitivity. During higher-frequency ultrasonication, a robust streaming effect combined with a cavitation effect may efficiently remove both pyritic and organic sulfur and increase coal desulfurization. Chemical leaching in combination with ultrasound can assist in reducing reagent usage, volume, and treatment time. Cracks on the coal surface are caused by the shockwave produced by ultrasonic leaching [63]. The leaching medium's shear force pushes the reagent to the centre of the coal particles. This effect increases chemical diffusion, resulting in a rapid mass transfer rate [63, 65-67]. The ultrasonic mechanism of coal processing is demonstrated in Figure 3 [63].

Acoustic streaming happens when sound waves produce unidirectional flow currents in a fluid. When ultrasonic energy is applied to coal slurry, two processes occur. The first is physical breaking, in which the chemical links between sulfur and coal are broken, and the second is leaching, in which a mass transfer process removes impurities. The primary objective of traditional coal desulfurization is to clean the surface of the coal matrix with little penetration into the core [62, 68-69]. Ultrasonic coal desulfurization is a well-known method for eliminating sulfur from the surface of the coal matrix [62]. According to one study, ultrasonic assistance enhanced sulfur desorbed [70-71] with 95% sulfur eliminated after nine minutes using PAA treatment and methanol extraction [72].

Flotation is an old separation method. Its high ash level and sulfur content made it inefficient with low-grade coals. Coal treated with ultrasound may overcome these drawbacks. Surface oxidation makes high-grade coal hydrophobic and low-grade coal hydrophilic. Ultrasound removes the hydrophilic group from oxidized coal, making it hydrophobic [28, 62]. Prolonged ultrasonic treatment is therefore not recommended due to the possibility of re-oxidation of the coal surface. Organic sulfur is challenging to remove by flotation due to its chemical reactivity. Introducing ultrasonic energy into the system increases the desulfurization of coal. Additionally, ultrasonic and electrochemical methods remove pyritic and organic sulfur from coal. It increases the mass transport of electrochemical species from the bulk solution to the electroactive surface during cavitation and streaming sonoelectrochemical coal processing. This increases electron transport and results in clean coal production during flotation [62].

Other research uses a mix of desulfurization methods. For example, Chinese high-sulfur coal was pre-treated with ultrasonics before being microwave desulfurized. Changes in ultrasonic treatment time and power should enhance the desulfurization rate, whereas increases in coal particle size should reduce desulfurization. Pre-microwave ultrasonic irradiation may enhance sulfur removal [23]. According to a study, demineralization using acids such as NaOH, KOH, or Na_2CO_3 followed by acid treatment with H_2SO_4 or HCl under ultrasonic assisted chemical leaching resulted in a higher degree of demineralization than non-ultrasonic leaching. Ultrasonic leaching with NaOH and 30% H_2O_2 resulted in the highest demineralization of all acid-leaching combinations tested. Ultrasonic desulfurization using 0.025 M NaOH at 30 °C eliminated 34% sulfur. It was previously believed that the NaOH diffused through cracks or coal pores because of cavitation and exposure to the localized sulfur site. 59% desulfurization of low-rank sub-bituminous Pakistani coal was obtained using the same process at a temperature of 30 °C, a sonication period of 1 hour and a concentration of 1 M NaOH. However, no data on the methods of sulfur reduction were reported [63].

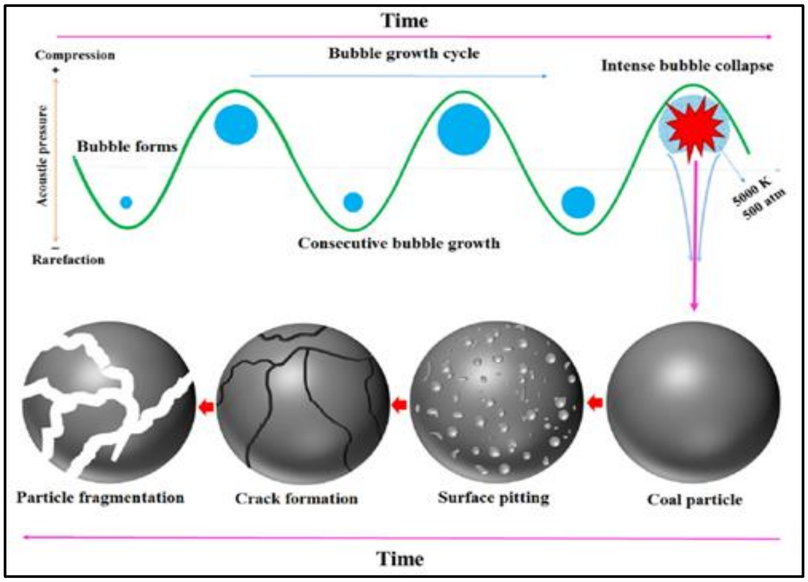

Figure 3: Ultrasonic mechanism of coal processing [63]

CONCLUSION

Finally, all methods have demonstrated the ability to remove high coal sulfur content. Additional variables, such as selecting proper parameter values that contribute to this capability, must be considered to achieve this capability. The use of combination pretreatment between acids and/or alkalis, as well as oxidation processes utilizing peroxyacetic acid in conjunction with microwave-assisted extraction and/or ultrasonic-assisted extraction, has become the preferred method for removing organic sulfur from coal in the last ten years or so.

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AUTHOR’S CONTRIBUTION

Syarifah Nursyimi Azlina Syed Ismail wrote and arranged the entire document. Siti Nur Lia Ali, Mohd Fauzi Abdullah, and Razi Ahmad contributed to the sections on "acid and alkaline leaching”, Mohd Azlan Mohd Ishak provided suggestions, and Nur Nasulhah Kasim formatted the articles.

CONFLICT OF INTEREST STATEMENT

The authors agree that this study was done without regard for self-interest, commercial purposes, or personal profit, and they declare that they have no competing interests with the funders.

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