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UPGRADING OF EAST KALIMANTAN LIGNITE CHARATERISTICS THROUGH INORGANIC SALT CHEMICAL ACTIVATION TREATMENT PRODUCING MICROWAVE ABSORBENT

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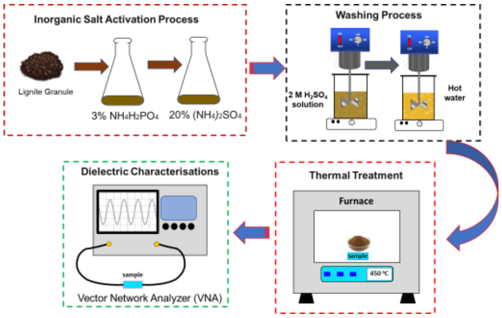
Graphical abstract



Abstract

The utilisation of microwaves in chemical processes is becoming increasingly popular due to its effectiveness over conventional methods. Certain processes necessitate microwave-absorbent materials, particularly when the target substrates exhibit inadequate dielectric properties. This study aims to investigate the transformation of East Kalimantan lignite into a microwave-absorbent material and identify the changes in its characteristics. Lignite samples were chemically treated using ammonium dihydrogen phosphate ($NH_4H_2PO_4$) and ammonium sulphate ($(NH_4)_2SO_4$) as activators, followed by washing and thermal treatment. These treatments converted lignite into a carbon-rich material with a chemical structure of activated carbon that closely resembling the structure of pure graphite. Notably, the fixed carbon content increased from 24.82% to 88.66%, indicating a significant enhancement in carbonisation. The dielectric loss tangent, initially undetectable, rose to 0.8898. Furthermore, the Fourier-transform (FTIR) spectrum revealed structural changes, including the transformation of alcoholic and aromatic ring groups, as well as alkane bonds, into an aliphatic ether structure.

Keywords: Chemical activation, dielectric loss tangent, lignite, microwave absorbent

Abstrak

Penggunaan gelombang mikro dalam proses kimia menjadi semakin popular kerana ia dianggap lebih berkesan daripada proses konvensional. Sesetengah proses ini memerlukan penyerap dibantu gelombang mikro kerana kekurangan sifat dielektrik bahan untuk dirawat. Penyelidikan ini bertujuan untuk memproses lignit, yang berasal dari Kalimantan Timur, menjadi penyerap gelombang mikro dan mengenal pasti perubahan ciri bahan tersebut. Lignit diproses secara kimia menggunakan $NH_4H_2PO_4$ dan $(NH_4)_2SO_4$ sebagai pengaktif berturut-turut diikuti dengan proses pencucian dan pemanasan. Rawatan ini menukar lignit kepada bahan berkarbon yang kaya dan membentuk struktur kimia asas karbon teraktif yang hampir sama dengan struktur grafit tulen. Beberapa ciri lignit utama telah berkembang menjadi penyerap gelombang mikro yang ditunjukkan oleh peningkatan karbon tetap daripada 24.8165% kepada 88.6608%. Tangen kehilangan dielektrik dinaikkan daripada tidak dapat dikesan kepada 0.8898. Sementara itu, spektrum FTIR menunjukkan perubahan struktur daripada kumpulan cincin alkohol dan aromatik serta ikatan alkana kepada struktur eter alifatik.

Kata kunci: Lignit, penyerap gelombang mikro, pengaktifan kimia, tangen kehilangan dielektrik

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1.0 INTRODUCTION

Lignite, commonly referred to as brown coal, is characterised by a low calorific value, typically below 5100 cal/g (adb), and high moisture, volatile matter, and ash contents [1]. These properties limit its widespread use as a fuel. However, Indonesia holds significant potential in this low-rank coal category. According to British Petroleum data, low-rank coal in Indonesia accounts for 29.4% of its total proven reserves, which amount to 39,891 million tons [2].

Numerous studies have investigated methods to improve the quality and utilisation of lignite. For instance, one study enhanced lignite's calorific value by adding low-sulphur waxy residue additives and heating the mixture at atmospheric pressure [3]. In another approach, researchers upgraded lignite via pyrolysis in a multi-stage fluidised bed reactor, producing gas, fuel, and char as end products [4]. Additionally, Zhang *et al*. (2020) developed a microwave-assisted pyrolysis technique using an synergism of exclusive catalysts (K_2CO_3, $CaCl_2$, and Fe_5O_4) to convert lignite char into syngas and semicoke [5]. Furthermore, raw low rank coal was subjected to low-temperature, high-pressure thermal treatment to enhance its suitability as fuel [6]. A recent study applied a leaching technique to reduce ash and sulphur contents in lignite [7].

Although lignite possesses unfavourable natural properties, it can be upgraded to activated carbon. Moreover, carbon-based materials, such as activated carbon and bio-char, serve as highly effective microwave absorbents due to their facile production or transformation via microwave heating. The ability of a material to absorb microwaves is quantified by its dielectric loss tangent (tan δ), which represents the ratio of the energy absorbed to the electrical energy that can be converted into heat energy within an electromagnetic field. Table 1 presents the tan δ values for various carbon materials [8].

Table 1 Dielectric loss tangent for some carbon materials at 2.45 GHz and 298 K

Carbon Material	tan δ = $(\varepsilon''/\varepsilon')$
Coal	0.02 – 0.08
Carbon foam	0.05 – 0.20
Charcoal	0.11 – 0.29
Carbon black	0.35 – 0.83
Activated carbon	0.57 – 0.80
Activated carbon [a]	0.22 – 2.95
Carbon nanotube	0.25 – 1.14
Csi nanofibers	0.58 – 1.00

[a] Activated carbon at a mean temperature of 398 K

Activated carbon or char, serving as microwave absorbents, has been widely employed across several applications. They have been used in microwave-assisted pyrolysis of oil palm shell waste biomass for bio-oil production [9], conversion of waste palm oil into diesel like fuel [10], and microwave pyrolysis of polystyrene and polypropylene mixtures to generate gasoline [11].

Several methods have been developed to demonstrate that lignite can be transformed into an effective microwave absorbent due to its enhanced dielectric loss tangent value. For example, a study of the dielectric properties of char derived from various ranks of coal, including brown coal, was conducted at high temperatures ranging from 850°C to 1600°C. Notably, the brown coal char heated at 1300°C exhibited a tan δ value of approximately 2.25 when measured at 2.45 GHz [12]. Another approach involved upgrading Indonesian lignite was via microwave irradiation at 1000 W in the presence of microwave susceptors. This method produced coke with a tan δ of approximately 0.0932 and 0.1435, depending on the use of activated carbon or graphite as susceptors, respectively [13]. Further investigations assessed the impact of variables such as heating temperature, particle size, initial moisture content, and microwave frequency on lignite's dielectric behaviour; the optimal dielectric loss tangent achieved was approximately 0.125 [14].

In this study, lignite was chemically activated using ammonium dihydrogen phosphate ($NH_4H_2PO_4$) and ammonium sulphate ($(NH_4)_2SO_4$) to produce an activated carbon suitable for microwave absorbent. These inorganic acidic salts facilitate pores development in lignite by cleansing surface impurities and disrupting carbon-carbon bonds. The selection of inorganic salts as chemical activators was to ensure that the activators could open the pores without compromising the integrity of lignite's inherently fragile bulk structure.

Following chemical activation, an additional thermal treatment was conducted to preserve the carbon structure and the newly formed pores and to eliminate non-carbonaceous components in gaseous form. As a result, the freed carbon atoms are irregularly arranged into elementary microcrystallites [15].

This study highlights the potential of lignite as a viable precursor for microwave absorbents, owing to its relatively high fixed-carbon content of 26.7% [16]. Furthermore, the developed treatment produced activated carbon with robust dielectric properties, making it a viable alternative to the commercially available activated carbon, as a microwave absorbent. Such advancements are particularly significant given the expanding use of microwave-assisted processes that depend on microwave absorbents materials.

2.0 METHODOLOGY

Lignite sourced from East Kalimantan coal mine was crushed and sieved using the ASTM standard sieve series to obtain 12-mesh size particles. The resulting material was statically immersed in a 3 wt% solution of $NH_4H_2PO_4$ (CAS number 7722-76-1) for nine hours. This was followed by immersion in a 20 wt% $(NH_4)_2SO_4$ (CAS number 7783-20-2) solution for equal duration. For both activators, the lignite-to-activator mass-to-volume ratio was maintained at 1:1.25 (m/v).

The activated particles then underwent a three-stage washing process. The first stage involved acid wash with a 2 M sulphuric acid (H_2SO_4, CAS number 7664-93-9) solution, followed by hot water rinse under gentle agitation for 90 minutes. The final stage was neutralisation rinse with water until the effluent pH reached neutrality.

The activated particles were subsequently subjected to a two-step heat treatment in a muffle furnace under ambient atmospheric conditions. The temperature was first ramped to 450°C and maintained for 30 minutes to initiate pore development during carbonisation. This was followed by a rapid increase to 950°C, held for 10 minutes to further enhance pore structure and stabilise the carbon matrix.

Finally, the lignite-activated carbon (LAC) sample was analysed to measure the dielectric loss tangent using a vector network analyser (VNA, Rohde & Schwarz ZVA24). Proximate and ultimate analyses were conducted according to American Society for Testing Materials (ASTM) standards. Proximate analysis was performed using a Sartorius ENTRIS124-1S balance, MEMMERT Oven UN 55, and Nabertherm B180 muffle furnace. Ultimate elemental analysis was determined with LECO CHN 2000 and LECO SC 632 instruments. Additionally, functional group identification was performed using an ABB FT-IR MB 3000 spectrometer. Finally, specific surface area was measured via the Brunauer, Emmett, and Teller (BET) method using a QuadraSorb Station 2 analyser.

A simple test was conducted to evaluate the efficacy of LAC as a microwave absorbent. In this test, 140 g of LAC was mixed with 200 mL of waste lubricating oil in a flask. The flask was equipped with a Type K thermocouple and a temperature controller calibrated to maintain the internal temperature at 400°C. The assembly was then placed in a domestic microwave oven and irradiated at 800 W for 120 minutes. Temperature readings were recorded every five minutes throughout the process.

3.0 RESULTS AND DISCUSSION

3.1 Characteristics Transformation

Table 2 indicates the characteristics upgrading of LAC, determined by proximate and ultimate analyses. Notably, these results demonstrate significant improvements in carbon content and dielectric properties.

Table 2 Lignite and LAC characteristics

Parameters	Lignite	LAC
Proximate Analysis (adb)		
Inherent Moisture (%)	38.4989	5.9063
Ash Content (%)	4.5120	2.3458
Volatile Matter Content (%)	32.1726	3.0870
Fixed Carbon Content (%)	24.8165	88.6608
Calorific Value (cal/g)	3.876	-
Surface Area (m²/g)	1.175	
Ultimate Analysis (adb)		
C (%)	56.05	88.35
H (%)	5.70	1.44
N (%)	0.89	0.95
O (%)	30.30	4.68
S (%)	0.88	0.85
Dielectric Properties at 2.45 GHz		
Dielectric Constant (ε')	-28.6483	0.5330
Dielectric Loss Factor (ε'')	-	0.4743
Dielectric Loss Tangent ($\tan \delta$)	-	0.8898

It can be observed that the initial characteristics of lignite include a calorific value (≤ 5100 cal/g), a low fixed-carbon content (24.82%), and high levels of moisture (38.50%), volatile matter (32.17%), and ash (4.51%). These suboptimal proximate properties result in an undetectable dielectric loss tangent, indicating minimal electrical energy absorption and negligible heat dissipation. Such behaviour is consistent with lignite's inherently low dielectric loss characteristics [15] .

The activation process reduced the moisture content to 5.91%. Ammonium sulphate ($(NH_4)_2SO_4$) acts as a dehydrating agent, binding water during the activation process [18]. The ash content also decreased following activation. This reduction is attributed to the use of a 20 % $(NH_4)_2SO_4$ solution—an acidic salt capable of dissolving mineral components—combined with a subsequent sulphuric acid wash that further removed inorganic residues. Meanwhile, the thermal stage reduced the volatile matter content to 3.09%. As a result of these combined treatments, the fixed-carbon content of LAC increased markedly to 88.66%, indicating successful conversion into a carbon-rich material.

Figure 1 illustrates the changes in proximate properties of LAC following treatment.

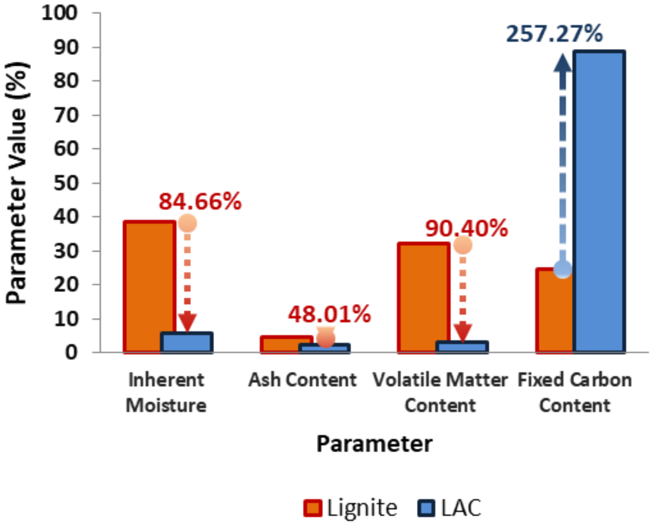
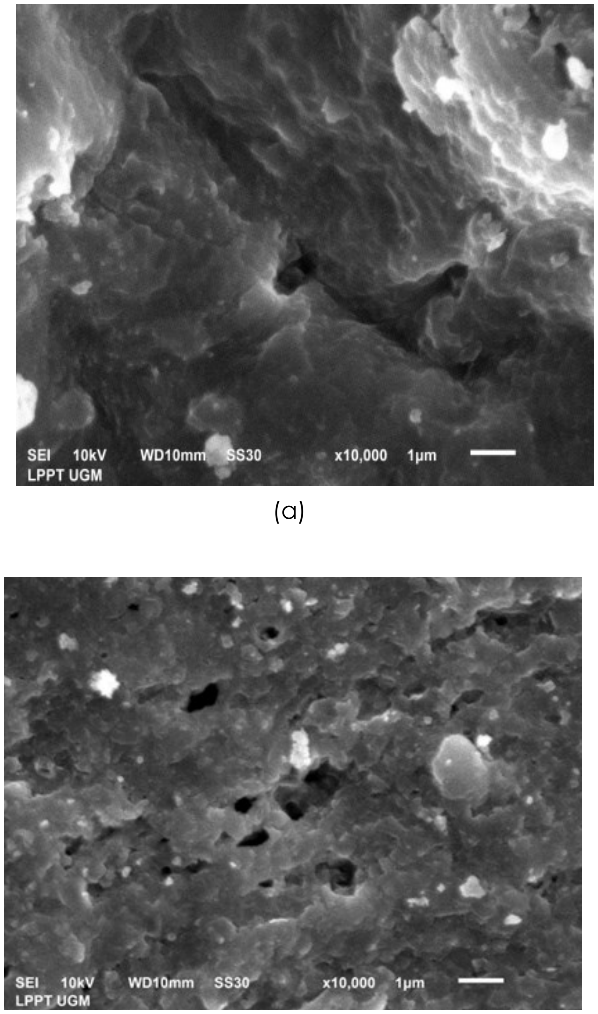


Figure 1 Percent change of proximate characteristics value

The percent change indicates the decreasing or increasing that compared to its initial value. Inherent moisture, ash, and volatile matter contents decreased by 84.88%, 48.01%, and 90.40%, respectively. Consequently, the fixed carbon content increased substantially by- 257.27%.

Table 2 presents the results of ultimate analysis, which reveal decreases in hydrogen and nitrogen contents, with a particularly decline in oxygen content, which is associated with a corresponding increase in carbon content. Li *et al.* (2020), reported that much of the oxygen in lignite is present in acidic functional groups, such as carboxyl (-COOH) [19]. The reductions in hydrogen and oxygen content in the LAC sample can be attributed to the removal of hydroxyl (OH) groups, through dehydration and dehydrogenation reactions, as well as the disruption of hydrogen bonding within the lignite structure [20].



(a)

(b)

Figure 2 Surface morphology images of (a) lignite; (b) LAC

Figure 2 displays surface morphology images of lignite and LAC, along with the increase in surface area from 1.175 m²/g to 104.74 m²/g, indicating the formation of a pore structure. Chemical activation facilitated pore opening by removing impurities and decomposing lignite's cellulose-based material, breaking carbon-carbon bonds and initiating carbonisation. Additionally, the thermal treatment further enlarged the pore sizes through the expulsion of volatile compounds.

Figure 3 illustrates the formation of pore structure in LAC, as evidenced by the greater N_2 adsorption volume compared to raw lignite. The N_2 adsorption isotherms conform to Type I curves based on IUPAC classification, characterised by a steep initial uptake at very low relative pressures (P/Po) range, followed by a gradual progression toward saturation at higher pressures. Type I isotherms are typical of microporous solids, such as activated carbons, which have relatively small external surfaces. Their performance is primarily governed by the micropore volume rather than the internal surface area [18,19]. The micropores development enables LAC to exhibit enhanced dielectric properties without compromising its fundamental carbon structure, maintaining its effectiveness as a microwave absorbent.

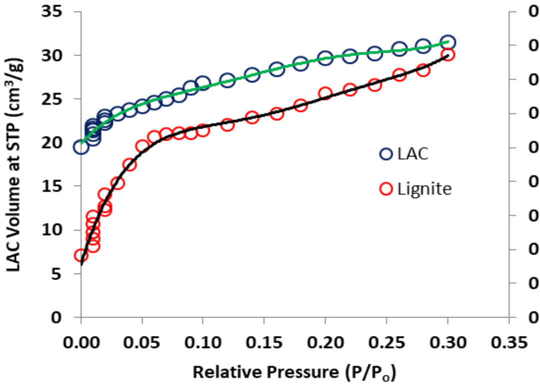


Figure 3 N_2 isotherm plot of lignite and LAC at 77.35 K

The efficiency of a material in generating heat under microwave irradiation is determined by three key dielectric parameters, namely the dielectric constant (ε'), dielectric loss factor (ε''), and dielectric loss tangent (tan δ). The ε' parameter quantifies the material's capacity to absorb electromagnetic energy, while the ε'' factor measures the amount of this energy converted into heat within the material [13]. The tan δ is the ratio of ε'' to ε', which reflects the efficiency of this energy conversion into heat. A higher tan δ value indicates greater microwave absorption and heat generation [10]. In this study, the ε' value of lignite and LAC increased from -28.6483 to 0.5330, demonstrating an improvement of microwave absorption capability. The negative ε' value observed in lignite indicates its very low absorption capability of electromagnetic energy. On the contrary, the material reflects electromagnetic waves wherein polarisation occurs in opposition to the applied electric field [20, 25]. Such negative dielectric constants are typically found in the MHz to THz frequency range, where the material is unable to synchronise with the fast oscillations of the field, resulting in phase lag and anomalous dispersion behaviour [26]. Moreover, the dielectric constant of coal generally diminishes as the frequency rises, particularly in low-rank coal varieties, where high moisture and ash contents can hinder the dielectric characteristic [27, 28]. As a result, the ε'' value for lignite could not be quantified, whereas the ε'' value for LAC was measured at 0.4743. This translated into a significantly improved dielectric loss tangent for LAC (tan δ = 0.8898), consistent with values typically observed for activated carbon.

Microwave irradiation is a promising and energy efficient heating technique, frequently utilised in chemical processes [29]. Since the microwave absorbent plays a crucial role in thermochemical processes, it is essential to evaluate LAC's effectiveness in raising temperature under microwave heating. An experiment was conducted using a domestic microwave operating at 800 W to heat waste lubricating oil (WLO), which being non-polar, cannot absorb microwaves directly. Therefore, LAC was added as a microwave absorbent. Figure 4 shows the temperature increase profile with the addition of LAC, compared to lignite.

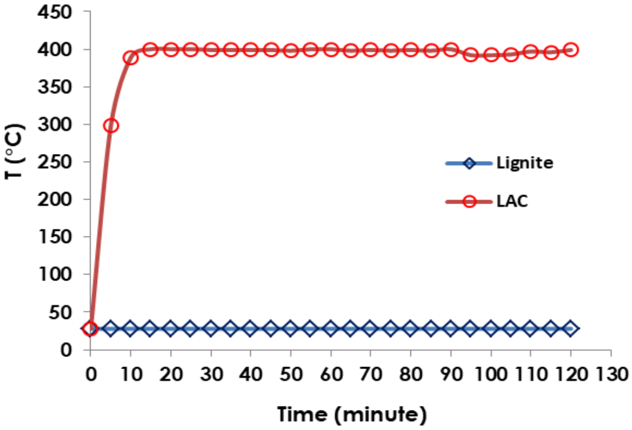


Figure 4 Raising temperature profile

The addition of LAC successfully raised the temperature to the target value of 400°C within 15 minutes, maintaining this level with an average accuracy of 99.49%. In contrast, the addition of lignite could not achieve the desired temperature and remained at room temperature. The carbonisation process established the fundamental chemical structure of activated carbon, which closely resembling the structure of pure graphite, characterised by carbon-carbon bonds [18,19]. This structure contains abundant π electrons and defect-free graphene areas, which facilitate efficient microwave absorption and heat generation [24].

All the analytical results support the conclusion that LAC functions as an effective microwave absorbent. Furthermore, this study demonstrates that raw lignite can be converted into a high-performance microwave absorbent via a relatively simple activation procedure

3.2 Functional Group Identification

FT-IR spectroscopy was employed to investigate the structural changes of raw lignite to activated carbon.

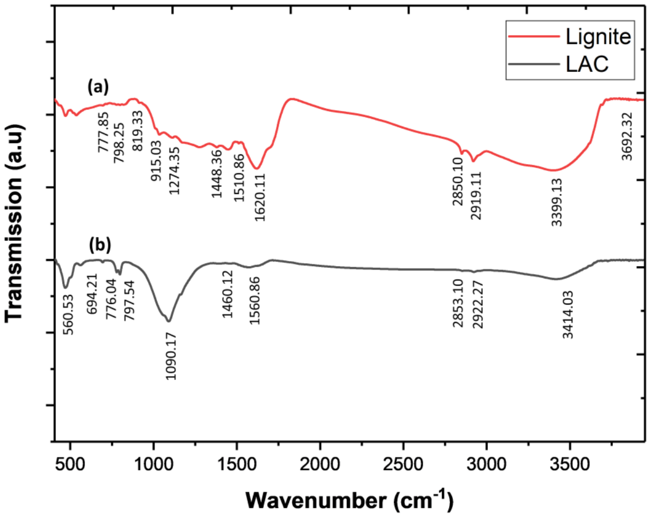


Figure 5 FT-IR spectra of (a) lignite; (b) LAC

Figure 5 exhibits the significant changes in absorption intensities in cross several spectral regions, with corresponding functional group assignments presented in Table 3 and Table 4.

Table 3 IR spectra interpretation of raw lignite

Spectrum Wavenumber (cm^{-1})	Spectrum Intensity	Interpretation
3692,32	Variable	Silanol
3399,13	Strong Broad	Phenolic
2919,11 2850,10	Variable	Alkyl/ Aliphatic
1620,11 1510,86	Strong Relatively Narrow	Aromatic Ring
1448,36 1274,35	Medium	Alkane Carboxylic Acid
915,03 819,33 798,25 777,85	Finger Print	C-H O-H C-C Bend

Table 4 IR spectra interpretation of lignite activated carbon

Spectrum Wavenumber (cm^{-1})	Spectrum Intensity	Interpretation	Compare to raw Lignite
3414,03	Broad Weak	Phenolic	Reduce
2922,27 2853,10	Variable	Alkyl	Reduce
1560,86	Variable	Aromatic Ring	Reduce
1460,12	Medium	Alkane	
1090,17	Strong Narrow	Aliphatic Ether	Increase
797,54 776,04 694,21 560,53		C-H O-H C-C Bend	

The FT-IR spectra of raw lignite indicate the presence of alcoholic functional groups, aromatic ring structures, and alkane bonds, observed at wavenumber ranges of 3500 cm⁻¹ – 3200 cm⁻¹, 1650 cm⁻¹ – 1550 cm⁻¹, and 1400 cm⁻¹ – 1300 cm⁻¹, respectively, as shown in Figure 5. Upon activation, these absorption bands decreased in intensity and shifted toward higher wavenumbers (1300 cm⁻¹–1050 cm⁻¹), with increased signal strength. This shift indicates the formation of an aliphatic ether structure. This structural change can be explained as follows: the immersion treatments with activators facilitated surface cleaning, dehydration, and the weakening of lignite's carbon bonds. Subsequent thermal treatment enhanced the removal of volatile components and increased microporosity, thereby enhancing the reactivity of the resulting char [33]. While volatiles were driven off, realignment of the aromatic regions occurred simultaneously. The rearrangement of the aromatic structure led to a reduction in pore volume. However, heating up to 900°C resulted in greater volatile loss than pore volume loss, leading to the overall expansion of the char structure.

3.3 Significant and Limitation of Study

Lignite is characterised by its weak structural integrity and low carbon content, leading to poor thermal stability and susceptibility to decomposition at high temperatures [26, 27]. To overcome these limitations, this study employed a chemical activation method combined with moderate-temperature heating, which effectively improved the dielectric loss (tan δ) properties, thereby improving lignite's suitability as a microwave absorbent. This approach offers an alternative to conventional physical activation methods, which typically require higher energy input and extreme temperatures [9, 10, 11].

From a scalability perspective, the proposed method demonstrates strong potential for industrial implementation. The process can be adapted into a continuous production system by incorporating integrated equipment such as a series of slow-mixing tanks, rotary filters, rotary furnaces, and rotary air coolers.

However, the use of chemicals during the activation stage raises environmental concerns, particularly the potential generation of hazardous waste and water contamination. To address these risks, several mitigation strategies are proposed: (i) selection of less hazardous activating agents, (ii) neutralisation of chemical waste prior to discharge to comply with environmental regulations; and (ii) regeneration and reuse of activated carbon to minimise waste and extend material life span.

In this study, the first strategy was implemented by using $NH_4H_2PO_4$ and $(NH_4)_2SO_4$, which are less toxic compared to the conventional activating agents. Future studies should explore the integration of full waste management systems and life cycle analysis to ensure environmental sustainability.

4.0 CONCLUSION

The increase in fixed carbon content from 24.8165% to 88.6608% demonstrates that the combination of chemical activation with inorganic salts and moderate-temperature heating effectively transformed lignite into activated carbon. Furthermore, the activated carbon derived from lignite shows potential as a microwave absorbent, as evidenced by its favourable dielectric properties, specifically a dielectric loss tangent value of 0.8898.

However, the use of multiple chemical activators may increase production costs and raise environmental concerns. Future studies should investigate the recyclability of these chemicals and develop strategies to minimise their environmental impact. Although the lignite's dielectric performance is promising, comparative analyses with existing commercial absorbents are necessary to assess its cost-effectiveness and scalability. Further research should focus on process optimisation and economic feasibility to support industrial application.

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Conflicts of Interest

The authors declare that there is no conflict of interest regarding the publication of this paper.

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