Enhancing Carbonaceous Char Derived from Cashew Shell Pyrolysis as a Renewable Coal Alternative

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ABSTRACT

Purpose: *This paper aims to evaluate the potential of carbonaceous char from cashew nut shell pyrolysis as a sustainable alternative to coal. By converting agricultural waste into a valuable energy resource, this research aims to address two critical challenges: effective waste management and the development of sustainable energy alternatives.*
Methodology: *The research methodology focused on converting cashew nutshell waste into a renewable energy source through a systematic process. Raw cashew nut shells were cleaned, dried at 60°C for 24 hours, and ground to a uniform size of 2-3 mm for consistent pyrolysis. The shells underwent pyrolysis in an oxygen-free environment to produce carbonaceous char, which was then chemically activated using phosphoric acid. After reacting at room temperature for two hours, the mixture was heated at 650°C in a muffle furnace to enhance the char's surface area and porosity. Post-activation, the char was washed with water and methanol, sonicated to remove impurities, and dried before being analysed using FTIR for structural changes. The ash content of the treated char was measured to evaluate its suitability as a coal substitute, ensuring precision throughout.*
Results & Findings: *The results of the study showed that the carbonaceous char derived from cashew nut shells exhibited significant improvements after undergoing chemical activation. The activation process using phosphoric acid (H_3PO_4) effectively increased the surface area and porosity of the char, which enhanced its adsorption properties. Fourier-transform infrared spectroscopy (FTIR) confirmed the presence of functional groups such as C=C, C=O, and C≡C, indicating successful structural modifications of the char post-activation. The ash content was reduced to 7%, a favourable result, as lower ash levels improve the char's industrial applicability. The char demonstrated improved combustion properties, making it a promising renewable substitute for coal.*
Originality/Value: *The originality of this paper lies in its innovative use of cashew nut shells, a widely overlooked agricultural waste, to produce carbonaceous char as a renewable coal substitute. By optimizing the chemical activation process with phosphoric acid (H_3PO_4), the study enhances the char's surface area and adsorption capacity, significantly improving its potential for industrial applications.*
Type of Paper: *Exploratory research*
Keywords: Sustainable energy, Bio-Waste utilisation, Sustainable energy, Waste management, Activated carbon, Cashew nut shell pyrolysis, Renewable energy resources, Chemical activation, Carbonaceous char, Renewable coal substitute.

1. INTRODUCTION :

Activated carbon (AC) is a versatile sorbent characterised by pore-rich combination, capable of removing contaminants such as heavy metals, pesticides, and organic pollutants from water and air. There is increasing research and innovation in bio-char production. (Chaiammart Nattapat et. al. (2024). [1]). While AC is traditionally produced from coal or wood, agricultural byproducts offer a sustainable alternative. Findings on AC has garnered significant notice from many scholars because of its broader applicability, including use as an absorptive medium, a capacitive layer, and a filtration material, among others. (Anas M. et. al. (2019) [2]). Activated carbons derived from cashew nut shells have been produced, activated and characterized through various processes like N_2 and CO_2. (Sadala D.V. and Mkayula L. L. (1995). [3]). Cashew nut shells (CNS) are abundant, renewable, and largely underutilized, with their disposal posing an environmental concern. Activated carbons derived from agricultural residues exhibit enhanced efficacy owing to their distinct and specialized characteristics. (Tangjuank. S. et. al. (2009). [4]). Activated carbon finds extensive use in various applications because of its high adsorption capacity in both gaseous and liquid phases. (Khezami Lotfi et. al. (2007). [5]). Innovative techniques such as microwave irradiation and hydrothermal carbonization are being advanced for the synthesis of activated carbon, offering promising prospects as ecologically sustainable methodologies. (Gaspard Sarra et. al. (2013). [6]). The conversion of agricultural residues such as rice husk, (Khan Nasehir E M Yahaya. et. al. (2010). [7]) empty fruit bunches, (Hameed B.H. (2009). [8]) coconut husk, (Tan I.A.W. et. al. (2007). [9]) and rice straw (Basta A.H., Fierro V., El-Saied H., Celzard A. (2009). [10]) into activated carbon not only mitigates the expenses associated with waste disposal but also transforms these materials into high-value products. In recent years, activated carbons are predominantly derived from coal, wood, or agro-based residues like coconut and palm shells, corncob, among others, through physical or chemical processes of activation. These methods of activation serve as a crucial means to enhance the porosity and adsorption capabilities of the resultant carbon materials. (Tangjuank S, Insuk N, Tontrakoon J, Udeye V. (2009). [11]). The concept of employing biomass from agricultural and livestock residues as a feedstock for the synthesis of activated carbons has garnered significant attention among researchers, particularly in nations with predominant agricultural practices. (Yee Jun Tham et. al. (2010). [12]). Cashew husk waste, constitutes approximately 67% of the unutilized fraction of cashew nuts, representing a significant portion of waste material. (Hunaidah et. al. (2019). [13]). Anacardium occidentale shell (ANS) contains cashew nutshell liquid (CNSL), which is extracted in the preliminary stages due to the release of harmful acidic vapours during shell combustion, posing risks to both public health and natural world (Cuillaume Kouassi Brou et. al. (2020). [14]). Various extraction techniques for CNSL have been documented in the literature, including CO_2-assisted extraction, ([15]) superheated steam treatment, ([16]) rapid roasting, ([17]) and solvent-based extraction. Each of these methods requires specialized equipment to ensure efficient extraction while minimizing environmental hazards. The choice of extraction method is often dictated by the desired quality of CNSL and its intended industrial applications, as it serves as a valuable feedstock in multiple sectors such as biofuels, lubricants, and polymer production. The cost of this adsorbent is heavily influenced by the availability and type of raw material sources. Consequently, the utilization of agricultural waste for activated carbon production is highly advocated, as it offers substantial economic benefits while simultaneously contributing to environmental conservation and sustainability. (Nguyen Hoc Thang et. al. (2021). [18]). Non-renewable energy sources are approaching the final stage of their life cycle, although occasional technological advancements may temporarily enhance access to these resources. (Furlan Claudia et. al. (2017). [19]). Utilizing these wastes for activated carbon production offers a dual advantage of reducing environmental waste and creating economically beneficial by-products. This study focuses on transforming CNS into activated carbon through pyrolysis and chemical activation, creating a high-value product from waste.

2. OBJECTIVES OF THE PAPER :

This paper aims to explore the feasibility of assessing the potential of cashew nut shell (CNS) in waste utilisation, for activated carbon production through various methods. The primary objective is to investigate and compare the efficiency of chemical and physical activation techniques in producing high-quality activated carbon. Additionally, the paper seeks to characterize the synthesized activated carbon using modern instrumentation such as FTIR and NMR to understand its chemical properties. The broader goal is to find sustainable alternatives to traditional coal-based carbon, contributing to waste management and cleaner energy solutions.

3. REVIEW OF LITERATURE :

Table 1: This provides a concise summary of the literature reviewed in this study, highlighting key areas of research related to activated carbon production and its applications.

S. No.	Area of Research	Focus and Outcome	Reference
1	Materials science.	Developing activated carbon electrodes.	Chaiammart Nattapat et. al. (2024). [1]
2	Production of activated charcoal.	Producing charcoal derived from cashew shell-based biomass by utilising nitrogen (N_2) as the activating material.	Anas M. et. al. (2019). [2]
3	Environmental science.	Methods to produce cashew shell-based charcoal and the liquid emanating from them.	Sadala D.V. and Mkayula L. L. (1995). [3]
4	Environmental and material science.	Efficacy of activated carbon derived from cashew nut shells in sorbing chromium (III) ions.	Tangjuank. S. et. al. (2009). [4]
5	Development of charcoal from renewable resources.	Producing activated carbon from thermo-compressed wood.	Khezami Lotfi et. al. (2007). [5]
6	Water quality management and pollutant removal technologies.	Production of activated carbon from rice husk.	Khan Nasehir E M Yahaya. et. al. (2010). [7]
7	Development and optimization of carbon materials	Optimizing the preparation conditions for producing activated carbons.	Tan I.A.W. et. al. (2007). [9]
8	Developing efficient carbon materials from biomass feedstocks.	Producing high-performance activated carbons from rice straw.	Basta A.H., Fierro V., El-Saied H., Celzard A. (2009). [10]
9	Production and optimization of carbon materials derived from agricultural by-products.	Impact of temperature of activation and heating time on the physical properties of activated charcoal.	Yee Jun Tham et. al. (2010). [12]

The study provides a solid foundation for producing and characterizing activated carbon from cashew nut shells and their liquid, but several opportunities for improvement and further exploration remain. One suggestion is to optimize activation methods by exploring advanced techniques like microwave or ultrasonic-assisted activation, which could enhance the performance and yield of the activated carbons. Integrating green chemistry principles, such as using environmentally friendly activation agents, would also enhance the sustainability of the process.

A key research gap is the limited exploration of various activation agents, which could potentially improve the traits of porous carbon. Moreover, there is a need to assess the economic feasibility and industrial scalability of the process, as well as the environmental impact through life cycle assessment. Comparative studies with other biomass sources would help determine the unique advantages of cashew nut shells, filling an important gap in the literature on sustainable activated carbon production.

4. RESEARCH METHODS :

4.1 Methodology:

(1) Treatment of Raw Cashew Shells:

The initial step in preparing activated charcoal from Anacardium nut shells involves thoroughly washing the shells. This process includes multiple rinses with water to eliminate any surface impurities or dust, providing an efficient method for cleaning. After washing, the shells are dried by exposing to heat in an oven at 60°C for about 24 hours. Once fully dried, the shells are ground into smaller fragments, typically around 2-3 mm in size.

(2) Refluxation and Distillation Process:

A quantity of 50 grams of finely ground Anacardium Nut Shell (ANS) was positioned in a 500 ml RB accompanied by 100 ml of toluene as a solvent. The mixture underwent reflux for 1 hour, during which the toluene facilitated the extraction of residual oils present in the shells post-combustion. After the reflux process, the mixture was filtered using a suction pump to separate the ANS char, which was subsequently desiccated at 50°C by morning.

The filtrate was then undergoing distillation using a hot plate set to 140°C and a magnetic stirrer to recover the toluene. The remaining residue in the round-bottom flask was analysed using infrared spectroscopy for characterization.

The distilled toluene was recovered and reused in subsequent refluxation cycles.

(3) Calculation of ash proportion of the char:

Residual ash can diminish the effectiveness of activated carbon, so it is desirable to have low ash levels in all activated carbon products. Water-soluble ash refers to the ash that can be removed through washing with water. Ash represents the non-combustible portion of coal, and the ash yield indicates the amount of material that will remain after combustion, which will require disposal.

In this process, the dried Anacardium Nut Shell (ANS) char was ground finely and heated at 60°C for 3 hours. Following this period, the finely ground carbon was transferred to a muffle furnace, initially set at 200°C for one hour. The temperature was elevated to 650°C to determine the ash content.

The char's ash content was examined through the measurement of ash and weight of the char sample,

% Ash= (Mass of the ash obtained ÷ Mass of the carbon residue taken) × 100

% Ash= (0.035g ÷ 0.5g) × 100 = 7

The ash fraction was established as 7% which indicates the effectiveness of the char to be used as a cost-effective catalyst by further modifying its structure.

(4) Activation of Bio-char:

To enhance the biochar's adsorption potential, the activation process is essential to increase void volume, pore channel size, and total surface. Heating the fine carbon powder expands its surface area, resulting in greater porosity.

Approximately 4 grams of the dried char, treated with toluene, was triturated and One part of the material was combined with 1.5 part of orthophosphoric acid, (Sivaraj Rajeshwari et. al. (2010). [20]). which acted as an activating agent. This was left undisturbed at RT for about 2 hours to facilitate the activation process. After the 2-hour activation period, the char was heated in an electric furnace at 650°C for 2 hours. Once cooled, the carbon was subjected to sonication with water twice for 15 minutes to wash away the orthophosphoric acid. The flushed carbon was cured at 62°C and sonicated again with 30 ml of methanol for 15 minutes. The methanol was subsequently eliminated accessing rotavapor, and carbon was washed with water before drying again.

The resulting dried carbon contained iron phosphate, which can be extracted using magnetic beads. Finally, the carbon was prepared for infrared spectroscopy analysis.

4.2 Reaction Using Synthesized Activated Char:

(1) PTA support on activated carbon:

Approximately 0.75 grams of carbon were accurately weighed and introduced into 40 mL of methanol, followed by continuous stirring. Subsequently, 0.25 grams of phosphotungstic acid, serving as an acid catalyst, were dispersed in 5 mL methanol and incorporated into the stirring mixture. The resulting solution was subjected to prolonged stirring on a magnetic stirrer for a duration of 12–16 hours.

Upon completion of the 12-hour stirring period, the alcohol was meticulously disposed of. The methanol-free activated carbon was then subjected to high-temperature processing in muffle kiln. at 250°C for 7–8 hours to facilitate complete dehydration and catalyst activation by eliminating any residual bound water molecules. After the thermal activation process, the carbon catalyst was recovered and employed in the esterification reaction between levulinic acid and ethanol to synthesize ethyl levulinate.

(2) Reaction work-up:

Approximately 0.5g of levulinic acid, 0.15g of PTA-activated carbon, and 5 mL of ethanol were introduced into a high-pressure reactor and subjected to reaction at an elevated temperature of 120°C for a duration of 6 hours. Due to the sluggish reaction rate at ambient conditions, the elevated temperature was employed to significantly accelerate the reaction kinetics.

Upon completion of the 6-hour reaction period, the heating was terminated, and the high-pressure reactor was cautiously withdrawn from the oil bath and set to cool to RT. Post-cooling, the reactor contents were filtered, using an excess of ethanol to thoroughly remove any residual solid catalyst. The resulting filtrate, containing a mixture of ethyl levulinate and ethanol, was then processed by rotary evaporation to efficiently remove the excess ethanol. This process yielded the final product, ethyl levulinate.

Yield data:

Amount of levulinic acid taken = 0.5g

Molecular weight of levulinic acid = 116.11 g/mol

Molecular weight of ethyl levulinate = 144.17 g/mol

Theoretical yield = (144.17 × 0.5) ÷ 116.11 = 0.6208g

Observed yield = 0.4025g

Percentage yield = (0.4025 ÷ 0.6208) × 100 = 64.83

Methodology overview

Fig. Ia

Fig. Ib

Fig. Ic

Fig. Id

Fig. Ie

Fig. If

Fig. Ig

Fig. Ih

Fig. Ii

Figure I

Figure 1 descriptions:

Fig. Ia. Dried Anacardium nutshell
Fig. Ib. Anacardium nutshell after grinding to 2-3mm mesh size
Fig. Ic. Refluxation of the Char with toluene
Fig. Id. Filtration
Fig. Ie. Distillation of residue obtained after filtration
Fig. If. Drying of toluene treated char
Fig. Ig. Mixing the char with activating agent
Fig. Ih. Heating the char in muffle furnace
Fig. Ii. Sonicate with water and methanol

Spectroscopic Characterization I:

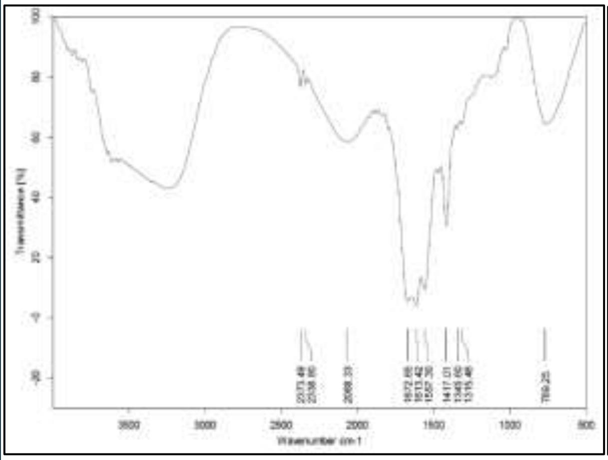

Fig. II (a)

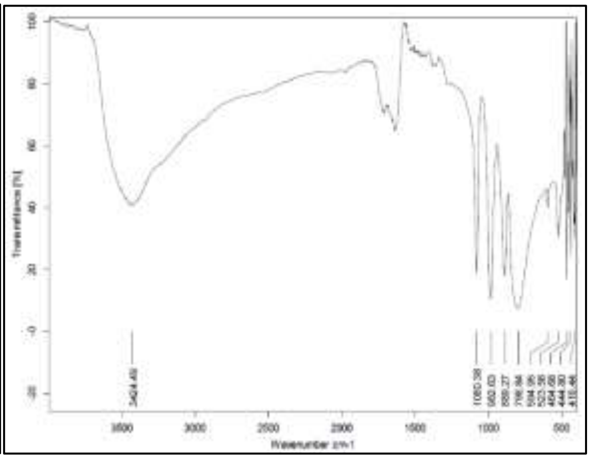

Fig. II (b)

Figure II

Figure 2 descriptions:
Figure II(a): FTIR Spectrum of activated charcoal
Figure II(b): FTIR spectra of PTA activated carbon

Spectroscopic Characterisation II:

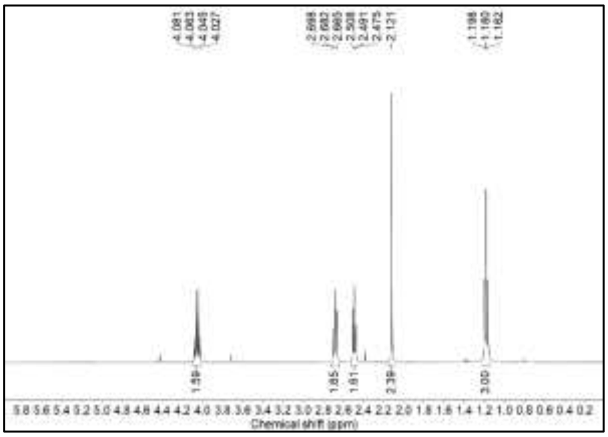

Fig. III (a)

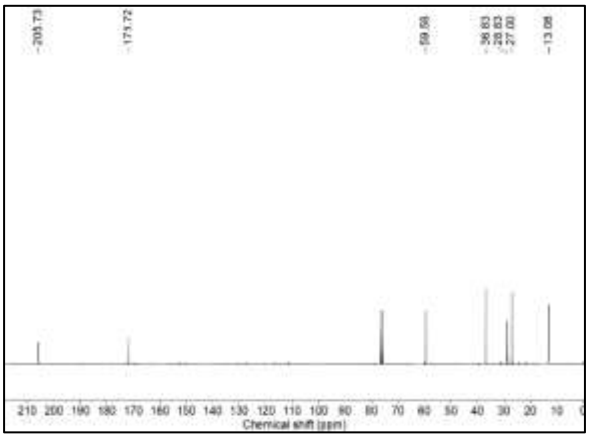

Fig. III (b)

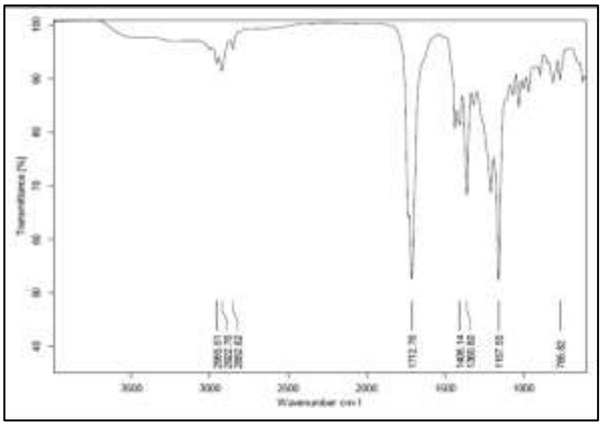

Fig. III ©

Figure III

Figure 3 Description:
Figure III (a): ^{1}H NMR Spectra of Ethyl Levulinate
Figure III (b): ^{13}C NMR Spectra of Ethyl Levulinate
Figure III (c): FTIR Spectra of Ethyl Levulinate

5. RESULT AND DISCUSSION :

The results indicate that chemical activation using orthophosphoric acid yields higher total area of the surface and volume of the void in the carbon derived from cashew nut shells compared to activation by physical methods. The temperature of activation and duration played critical roles in determining the adsorption capabilities of the produced activated carbon. FTIR analysis verified the existence of functional groups like C=C and C=O, essential for adsorption properties. NMR spectra revealed significant structural consistency across batches. The synthesis of ethyl levulinate using PTA-activated carbon as a catalyst was successful, achieving a yield of 64.83%. This underscores the potential of CNS-derived activated carbon in industrial catalytic processes.

The comparison between chemical and physical activation methods revealed that chemical activation, specifically using orthophosphoric acid, produced activated carbon with superior pore structure and surface area. Physical activation, while simpler, resulted in lower adsorption capacity, possibly due to incomplete removal of tarry substances during carbonization. The ash content of the final product, calculated at 7%, is within acceptable limits for industrial applications, suggesting its potential for use in water purification and other filtration processes. Additionally, the TLC analysis confirmed the purity of the synthesized ethyl levulinate, with an R_f value of 0.45, indicating minimal impurities. These results validate the effectiveness of using CNS for the generation of high-performance activated charcoal, with practical applications in both environmental and industrial processes.

The results from this study align with existing literature on biochar activation, particularly in terms of the increased surface area and adsorption capacity. This study further validates that chemical activation, especially using phosphoric acid, results in more effective carbon structures for industrial applications. Previous research has also demonstrated that agricultural waste-derived activated carbon, such as from rice husk and sugarcane bagasse, holds significant potential. Our findings on cashew shell pyrolysis reinforce the notion that bio-waste can contribute to sustainable energy solutions and reduce dependency on fossil fuels.

6. SUGGESTIONS/RECOMMENDATIONS BASED ON THE ABOVE ANALYSIS :

(1) **Optimization of Activation Methods**: Future research should focus on optimizing the chemical activation process by experimenting with different acid concentrations and activation temperatures to further enhance the adsorption properties.

(2) **Scale-up Potential**: Given the promising results on a laboratory scale, scaling up the production of cashew shell sourced activated carbon material is recommended. This might be an economically viable solution for industries seeking sustainable alternatives to traditional carbon sources.

(3) **Broader Applications**: The successful catalytic conversion of levulinic acid to ethyl levulinate suggests that CNS-derived activated carbon can have broader applications, including in biofuel production. Further studies could explore its use in other catalytic reactions and environmental cleanup processes.

7. CONCLUSION :

This study demonstrates the effective conversion of cashew nut shells into high-quality activated carbon through chemical activation using orthophosphoric acid. This approach offers a sustainable solution for both waste management and carbon production, potentially reducing reliance on fossil fuels. The implementation of the synthesized carbon as a catalyst in esterification reactions further highlights its versatility. Further research on optimizing the activation process and exploring broader applications of CNS-derived activated carbon could substantially contribute to sustainable industrial practices. The project also provided valuable hands-on experience with various chemical processes and analytical techniques, enhancing both practical knowledge and environmental awareness.

This study successfully demonstrates the feasibility of utilizing cashew nut shells, an agricultural waste product, for the production of carbon black. Activation by chemical method using orthophosphoric acid proved to be more effective than physical methods, yielding activated carbon with enhanced surface and free volume, which is critical for adsorption applications. The use of CNS-derived activated carbon in catalytic reactions, such as the synthesis of ethyl levulinate, further underscores its industrial relevance. The low ash content and high purity achieved in the final products highlight the potential of this sustainable alternative for various applications, including environmental remediation and industrial catalysis.

This study confirms that CNS, a plentiful farm residue, can be effectively altered into high-surface-area carbon through chemical activation processes. The high surface area, low ash content, and enhanced adsorption properties of the resulting char make it a strong candidate for replacing coal in industrial applications. Additionally, the application of this biochar in catalytic processes highlights its versatility beyond energy production, offering potential for environmental remediation. Future work should focus on scaling up production and exploring additional applications in biofuel and water purification systems.

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