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RESEARCH ON THE PREPARATION, PHYSICO-CHEMICAL PROPERTIES AND **APPLICATION ORIENTATION OF MODIFIED** CARBON MATERIALS FROM STRAW

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INTRODUCTION

Rice is one of the oldest crops, first grown thousands of years ago. Today, rice is grown in more than 100 countries and is consumed by more than half of the world's population as a staple food . The annual worldwide rice production is about 700 million tons of rice, of which 95% is produced in Asia. In Vietnam, the average annual rice production is about 40 million tons. Straw is one of the main by-products of rice farming. If an average of one ton of rice produces 1 - 1.2 tons of straw, with current rice production, it is estimated that the amount of straw waste can be more than 40 million tons/year, accounting for about 62% of the biomass of agricultural waste. . This amount of straw is often burned in the field, polluting the environment or used as fuel, as feed in livestock, growing mushrooms, etc. with low economic value. Moreover, the combustion process will release into the atmosphere substances such as CO₂, CO, CH₄, NO_x, SO₂, aromatic hydrocarbons... These substances cause air pollution and seriously affect the environment. human health. While the straw component contains about 0.6 % N; 0.1% P; 0.1% S; 1.5% K; 5% Si; 40% C, as a carbon-rich source of raw materials, has the potential to replace dwindling fossil fuels. Therefore, recently this source of by-products has been interested by scientists in research and orientation for the production of biofuels, as materials for adsorbing pesticides, dyes, oil spills and heavy metal ions, and making electrode materials for batteries or supercapacitors.

In addition, rice husks are also an abundant and low-cost source of biomass waste during rice production. Annually, the world generates about 140 million tons of rice husk, while in Vietnam it is estimated that the average generation is over 8 million tons. Fuel from rice husk is used a lot in daily life (cooking, cooking for animals) and production (making bricks, drying rice). After burning, the organic components will be converted into ash, in which the silica has a mainly amorphous structure, accounting for the highest percentage by volume, about 80-97%. SiO₂ from rice husk ash has a large specific surface area and high porosity, good chemical stability and high strength, so it has great potential as an adsorbent to remove fatty acids and pigments in the process. refining vegetable oils, heavy metals, pesticides, dyes, other organic pollutants from wastewater or used as electrode materials.

In recent years, domestic and foreign scientists have been studying the use of carbon sources from biomass waste in general and straw in particular to make carbon materials for application as adsorbents and electrical materials. pole. In addition, the conversion of rice husk ash into SiO_2 for application as electrode materials is believed to be an economic and sustainable direction. In order to integrate with the general trend of the world on the issue of finding new sources of raw materials with high economic value, the research and manufacture of materials from the abundant waste straw and rice husk ash makes an important contribution to the construction a sustainable agriculture, protecting ecological balance is an urgent and practical issue. However, up to now, there have not been many detailed studies on converting rice straw and rice husk ash into adsorbent materials to treat water pollution and electrode materials for Li batteries or supercapacitors. From this fact, we carry out the thesis topic: Research on preparation, physico-chemical properties and application orientation of modified carbon materials from straw. The objective of the thesis is i) Research on the preparation of carbon materials from waste straw and rice husk ash; ii) Study the physico-chemical properties of the obtained carbon materials and iii) Study the applicability of the obtained carbon materials as adsorbents and electrode materials.

This thesis is carried out with the desire to contribute to solving two problems in our country today, the risk of environmental pollution of straw and increasing the use value of straw and rice husk ash by-products. The results of the thesis are the scientific basis that opens the direction of using straw and rice husk ash in the preparation of application-oriented carbon materials as adsorbents and electrode materials, thereby expanding to other applications. other biomass waste sources.

CHAPTER 1: OVERVIEW

1.1. Overview of carbon materials derived from plant biomass *1.1.1. General introduction to carbon materials from plant biomass*

Lignocellulose plant biomass is the plant dry matter that can be considered the most abundant material on earth. Currently, lignocellulosic materials are the only renewable resource that contains a carbon source that can be converted into products in solid, liquid and gaseous forms. Due to depletion of crude oil reserves and increasing global energy consumption, many countries are relying on carbon-based biomass as an alternative source for fuel production and chemical industry. Lignocellulosic biomass can be classified into (1) agricultural waste that arises mainly from various agricultural and farming activities, (2) energy crops grown to produce biofuels or electricity, and (3) forestry residues from logging areas and management practices.

lignocellulose biomass consists of 35-55% Most cellulose, 20-40% hemicellulose and 10-25% lignin. The composition varies according to the type of biomass, the locality, the climatic conditions and the soil in which it grows. The structurally stable lignocellulosic biomass prevents the hydrolysis and fermentation of biomass by solvents and microorganisms, which is a challenge for the efficient use of the components present in the biomass. Therefore, a pretreatment stage is often required to break down the complex structure of the biomass and thereby increase the recovery of the components and ensure the economic feasibility of the bioconversion. . Over the years, various pretreatment methods such as autolysis, hydrolysis in dilute acids, alkalis, inorganic salts, steam explosion and ionic liquids have been scrutinized by researchers.

In Vietnam, the potential source of biomass is more than 99 million tons per year, of which the Mekong River Delta accounts for 33.4 % of that total. The Mekong Delta region has great potential to use biomass electric energy from agricultural by-products such as rice straw, rice husks, bagasse, corn stalks and cattle manure, as they are abundantly produced in this area. Therefore, the development of renewable energy from agricultural by-products in Vietnam and the Mekong Delta has high potential. In addition, there are many research works taking advantage of Vietnam's abundant agricultural byproducts as adsorbents to treat polluted water environment.

1.1.2. Methods to prepare carbon materials from plant biomass

- 1.1.2.1. Physical method
- 1.1.2.2. Chemical method
- 1.1.2.3. Hydrothermal carbonization (HTC) method
- 1.2. Some common uses of straw and rice husk ash

1.2.1. Common uses of straw

- 1.2.1.1. Use in agriculture
- 1.2.1.2. Energy regeneration
- 1.2.1.3. Paper and pulp production
- 1.2.1.4. Production of adsorbents for environmental control

1.2.2. Common applications of rice husk ash

- 1.2.2.1. Production of adsorbents for environmental control
- 1.2.2.2. Building materials
- 1.3. About methylene and arsenic blue dyes
- 1.3.1. Introduction of methylene blue dye

1.3.2. General introduction about arsenic

- 1.3.2.1. The form of arsenic in nature
- 1.3.2.2. Arsenic toxicity
- 1.3.2.3. Arsenic pollution situation in Vietnam

1.4. Theoretical basis of the adsorption of MB dyes and metal anions As on biochar

1.5. Overview of electrochemical sources

- 1.5.1. Introduction to electrochemical sources
- 1.5.2. Rechargeable Li-ion Battery
- 1.5.3. Super Capacitor
- 1.5.4. Classification and development trend of supercapacitor
- 1.5.4.1. Electrochemical double layer capacitor
- 1.5.4.2. Fake capacitor
- 1.5.4.3. Hybrid capacitors
- 1.6. Research status related to the topic
- 1.6.1. Biochar material from biomass modified by salts
- 1.6.2. Magnetic biochar from biomass applied as adsorbent
- 1.6.3. Modified activated carbon applied as electrode material
- 1.6.4. Silica in electrochemical capacity enhancement
- **1.7.** Conclusions drawn from the review

From the synthesis of the research situation related to the above topic, it shows that the study of using carbon sources from biomass waste in general and straw in particular to make carbon materials applied as adsorbents for environmental treatment. water field and electrode materials in energy storage technology have obtained positive results. However, no work has been found to study the preparation, physico-chemical properties and applicability of carbon materials from straw, especially lignin from straw, applied as an adsorbent to treat arsenic metal, Color dyes and modified carbon materials N, Mn, Si (SiO₂ from rice husk ash) are clearly and systematically applied as electrode materials. Moreover, to our knowledge, this is the first time in Vietnam that research on synthesizing carbon materials from straw by hydrothermal method and its

application. Therefore, this thesis topic implements the following research contents:

- Preparation and investigation of MB adsorption capacity of biochar by hydrothermal carbonization method from straw.

- Prepare and investigate the adsorption capacity of MB, As(V) and As(III) of magnetic biochar by hydrothermal carbonization method from lignin extract of straw.

- Preparation of activated carbon modified by N by pyrolysis of lignin extract of straw; Preparation of porous SiO₂ from rice husk ash by dissolution-precipitation method. Investigation of the electrochemical properties of activated carbon modified by N, with and without the combination of SiO₂.

- Preparation and investigation of electrochemical properties of N and Mn co-modified activated carbon by pyrolysis of lignin extract of straw.

CHAPTER 2: EXPERIMENT

2.1. Raw materials and chemicals



Figure 2.1. Schematic summary of the experimental procedure

- 2.2. Preparation of adsorbent
- 2.2.1. Preparation of biochar (BC)













Figure 2.6 . Process of preparing SiO 2 from rice husk ash

2.4. Investigation of the adsorption capacity of BC

The MB adsorbent was selected to investigate the adsorption of BC material samples. Experiments were carried out by stirring (500 rpm) 0.1 g of the material powder and 100 mL of MB solution with the desired initial concentration at room temperature. After the exposure time was determined, the BC adsorbent was separated by centrifugation for 5 min (3000 rpm) and the liquid fraction was diluted as necessary to determine the remaining concentration of MB solution.

2.4.1. MB solution adsorption capacity of BC. samples

To compare the adsorption capacity of BC product samples, the conditions of the adsorption process were fixed as follows: MB concentration 90 mg/L, adsorption time was 90 minutes.

2.4.2. Effect of contact time and adsorption kinetics of BCZn samples2.4.3. The adsorption isotherm of BCZn

2.5. Investigation of the adsorption capacity of MC materials

Three pollutants were selected as adsorbents: MB, As(V) and As(III). All experiments were performed by stirring (500 rpm) 0.1 g of material powder in 100 mL of MB, As(V) and As(III) solutions respectively with time, concentration, suitable temperature and pH. The initial pH value was adjusted with HCl or NaOH solutions to achieve the desired value. After the contact time has been determined, the MC adsorbent is separated by magnetization and the liquid fraction can be diluted as necessary to determine the concentration of MB or As(V) or As(III) solution. remaining at that time.

2.5.1. MB solution adsorption capacity of MC samples

To compare the adsorption capacity of MC product samples, the conditions of the adsorption process were fixed as follows: Initial MB concentration was 100 mg/L (pH \sim 7), adsorption time was 90 minutes at room temperature.

2.5.2. MB, As(V) and As(III) adsorption capacity of MC-1.0

- 2.5.2.1. Effect of contact time and adsorption kinetics
- 2.5.2.2. Effect of concentration and adsorption isotherm

2.5.2.3. Effect of pH of the first solution

- 2.5.2.4. Effect of temperature and adsorption thermodynamics
- 2.5.3. Evaluation of the reusability on the MC-1.0
- 2.6. Electrochemical analysis of ACN, ACN/SiO2 and ACNMn
- 2.6.1. Fabrication of electrode film
- 2.6.2. Capacitor installation process
- 2.6.3. Cyclic potential scanning method
- 2.6.4. Fixed current discharge measurement method
- 2.6.5. Electrochemical impedance method
- 2.7. Characteristic analysis of materials
- 2.7.1. X-ray Diffraction
- 2.7.2. X-ray energy dispersive spectrum
- 2.7.3. Furier transform infrared spectrum
- 2.7.4. Raman scattering spectra
- 2.7.5. Scanning electron microscopy
- 2.7.6. Transmission electron microscopy
- 2.7.7. Isothermal adsorption desorption N₂
- 2.7.8. Determination of zero charge point pH_{PZC}

CHAPTER 3 : RESULTS AND DISCUSSIONS

3.1. Chemical composition of straw and rice husk ash

The analysis results of straw material composition showed that the carbon content in the original straw sample was quite high (49.16 %) and the three main polymers content was cellulose (36.83 %), hemicellulose (27.18 %)), lignin (17.2 %). At the same time, the raw material composition of rice husk ash in the initial ash sample was mainly SiO_2 (85.40 %). This result confirms that the composition of the materials is in the normal range of rice straw and rice husk ash, which proves that this is a suitable source of raw materials for the application of carbon and silica materials.

3.2. The results of the BC modulation process

3.2.1 . Effect of salt denaturing agent on properties of BC

The results showed that the addition of salt, especially ZnCl₂ in the HTC process supported the carbonization of the straw, which coincided with previous studies when adding salt in the HTC glucose process as well as some biomass. other products such as corn cob, pine wood, etc. ZnCl₂ modified biochar has a porous structure, specific surface area and large pore volume, which is the basis for use as an effective adsorbent.

STT	Sample	Modulation efficiency, %	Adsorption efficiency, %	
1	RS	-	38.76	
2	BC-0	66.1	62.58	
3	BCNa	58.4	77.60	
4	BCCa	55.8	84.27	
5	BCZn	52.7	91.14	

Table 3.3. Preparation and adsorption efficiency of BC samples



Figure 3.1. Photos of samples (a) RS, (b) BC-0, (c) BCNa, (d) BCCa and (e) BCZn



Figure 3.4. SEM images of samples RS (a) and BCZn (b)

3.2.2. Evaluation of MB adsorption capacity on BCZn samples

3.2.2.1. Effect of contact time and adsorption kinetics



Figure 3.5. Effect of contact time on adsorption capacity at different MB concentrations of BCZn samples



The kinetics of the adsorption process followed the appare 2nd order model. The adsorption occurred rapidly and almost reached equilibrium after 30 min.

3.2.2.2. Adsorption isotherm

The isotherm of the adsorption process is consistent with the Langmuir model. The maximum adsorption capacity reached 86.55 mg/g, which can compete with many previously reported adsorbents. Thus, from the waste straw, biochar has been prepared by a simple, low-cost method with good adsorption capacity in wastewater treatment.

3.3. MC modulation results

3.3.1. Effect of the concentration of FeCl₃ on the properties of MC



Figure 3.7. XRD of MC and FO





Figure 3.11. Adsorption - desorption curve (a) and pore size





Figure 3.12. TEM (a) and SEM (b) images of sample MC-1.0

With a concentration of 1 M $FeCl_3$, the magnetic biochar has a porous structure, high magnetization, which is a prerequisite for high adsorption and regeneration capacity.

3.3.2. Evaluation of the adsorption capacity of MB, As(V) and As(III) on MC-1.0. sample

3.3.2.1. Effect of contact time and adsorption kinetics



Figure 3.14. Effect of contact time on adsorption capacity at different concentrations of MC-1.0 sample

The kinetics of the adsorption process follows the apparent 2nd order model. MB adsorption occurs rapidly and almost reaches equilibrium after 30 min , while that for As(V) and As(III) is about 75 minutes exposure.

3.3.2.2. Effect of concentration and adsorption isotherm

The isotherm of the adsorption process is consistent with the Langmuir model . The maximum adsorption capacity achieved is 110.64 mg / g (MB), 2.28 mg/g (As(V)) and 2.03 mg/g (As(V)), which can compete strongly with many previously reported adsorbents.



Figure 3.16. Experimental data and adsorption isotherms according to Langmuir and Freundlich models on MC-1.0

3.3.2.3. Effect of solution pH of MB, As(V) and As(III)

The pH_{PZC} of MC-1.0 is about 6.32. At pH 7, electrostatic attraction occurs between the negative active sites on MC-1.0 and the MB cation ion at higher pH, facilitating more favorable adsorption. Meanwhile, the most favorable pH for As(V) adsorption is 7 and As(III) is 9 on MC-1.0 material. With H $_3$ AsO $_4$ (pKa $_1$, pKa $_2$ and pKa $_3$ are 2.1; 6.7 and 11.2 respectively and H₃AsO₃ (pKa₁, pKa₂ and pKa₃ are 9.1, 12.1 and 13.4), the As adsorption capacity of magnetic biochar depends mainly on two factors electrostatic interaction and surface complexation.

3.3.2.4. Thermodynamics of adsorption

Negative values of $\Delta G \sigma$ indicate that the MB, As(V) and

As(III) adsorption processes on MC-1.0 are spontaneous and thermodynamically favorable . This adsorption of MB is mainly physical, while the adsorption of As(V) and As(III) on MC-1.0 is physical and partly chemical at the same time. The positive values of ΔS° *indicate the* MC's affinity for MB and As increases the randomness at the solid/liquid interface during adsorption.

Adsorbed	Temperature	ΔS^{θ}	ΔH^0	ΔG^0	
substance	(K)	(J/mol K)	(<i>kJ</i> /mol)	(kJ/mol)	
	303			-5.38	
MB	313	67.41	15.06	-6.01	
	323			-6.73	
	303			-4.70	
As(V)	313	89.10	22.32	-5.51	
	323			-6.49	
	303				-2.95
As(III)	313	81.16	21.67	-3.68	
	323			-4.57	

Table 3.11. Thermodynamic parameters of adsorption on MC-1.0 at different temperatures

3.3.2.5. The ability to regenerate

After five cycles, there was only a slight decrease in elimination from 107.32 to 98.73 mg/g for MB, from 2.165 to 1,992 mg/g for As(V) and from 1,907 to 1,755 mg/g for As(III). All have over 90% usability after five cycles, indicating that MC-1.0 is highly regenerative in application to handling MB, As(V) and As(III).

Thus, by simple preparation method and cheap raw materials, it is possible to prepare magnetic biochar that adsorbs MB, As(III) and As(V) well, easy to recover by external magnetic field, promising promises a line of high economic efficiency materials in the treatment of polluted water environment.

The specific surface area BET of SiO ₂ obtained is quite large (258.3 m²/g). This is in agreement with the results obtained from SEM and TEM. The SEM and TEM images show that the SiO₂ particles have a fairly uniform distribution, moreover, the TEM images show that the size of the microcrystalline particles is about 10-15 nm, creating the porous structure of the material. This is an important feature that makes SiO₂ material separated from rice husk ash with high porosity applied in electrochemical electrodes.

3.5. Result of the preparation of N modified activated carbon *3.5.1*. *Effect of urea amount on properties of ACN samples*

Samples have amorphous carbon structure with some graphite crystals, the degree of crystallization of the product decreases with increasing urea doping rate. Sample ACN-0.05 has many advantages in terms of pore area and size for ion diffusion in use as electrode material.



Figure 3.27. Adsorption-desorption curve (a) and pore size distribution (b) of ACN



Figure 3.28. SEM images of samples AC (a) and ACN-0.05 (b)

3.5.2. Investigation of electrochemical properties of ACN samples



Figure 3.29. a) CV curve at 5 m/s scan speed and b) Specific capacitance at different scan rates of ACN samples



Figure 3.30. a) GCD curve at 0.5 A/g current density and b) EIS spectra of the ACN samples

The survey results show that the ACN samples have the characteristics of EDLC and good electrochemical reversibility. When increasing the content of N doping, the specific capacitance of the supercapacitors tends to increase, indicating

that the N heteroatoms in the structure have improved their electrochemical performance by improving the diffusivity associated with the base . imitation capacitor . In which, sample ACN-0.05 has the highest specific capacitance (169.3 F/g), stability, good diffusivity and relatively low impedance, which is the most suitable for application as electrode material. for supercapacitors.





Figure 3.34. a) GCD curve at 0.5 A/g current density and b) EIS impedance spectra of the ACNSi samples

The addition of SiO $_2$ can increase the charge transfer from the electrolyte solution to the electrode surface, but also increase the charge transfer resistance. By simple ball grinding

method, which is easy to control the SiO_2 content, the obtained material has a relatively high capacitance (~182 F/g), a long charge-discharge time, and offers great potential in many applications. electrode material.

3. 6. ACNMn . modulation results 3.6.1. Effect of KMnO4 ratio on properties of ACNMn samples

The MnO particles are uniformly dispersed on the product surface. Sample ACNMn-1:0.02 has high crystallinity, specific surface area and large pore volume, which shows advantages in improving the electrochemical performance of the product.



Figure 3.37. Adsorption–desorption curve (a) and pore size distribution (b) of ACNMn . samples



Figure 3.38. SEM images of sample (a) ACN-0.05, (b) ACNMn-1:0.02 and (c) EDS mapping image of C, O, Mn elements of sample ACNMn-1:0.02

3.6.2. Investigation of electrochemical properties of ACNMn samples



Figure 3.45. GCD 0.5 A/g current density and b) EIS of ACNMn samples

Among the ACNMn electrode samples, the ACNMn-1:0.02 sample gives the highest specific capacitance (nearly 200 F/g) and the longest charge-discharge retention time, while also having good diffusion capacity and relatively low impedance. exhibits outstanding electrochemical performance.

CONCLUSIONS AND RECOMMENDATIONS

Conclusions

From the obtained results of the thesis, the following conclusions can be drawn:

1. Successfully prepared biochar material from straw waste by one-stage hydrothermal method in the presence of different salts, in which ZnCl 2 salt is the $_{most}$ suitable for carbonization and MB pigment adsorption. The maximum MB adsorption capacity of the product reached 86.55 mg/g.

2. Successfully prepared magnetic biochar from straw by two-stage hydrothermal method with different concentrations of FeCl _{3 solution}. The prepared material has high specific surface area and high magnetization. The maximum adsorption capacity was 110.64 mg/g MB, 2.28 mg/g As(V) and 2.03 mg/g As(III).

3. Successfully synthesized SiO $_{2 \text{ material}}$ by dissolution - precipitation method with rice husk ash as the starting material. The obtained SiO $_{2}$ material has a crystalline phase structure, relatively uniform particles distribution (about 10 - 15 nm) and large specific surface area (258.3 m 2 /g), suitable for use in electrode material.

4. Successfully prepared N and Mn modified activated carbon from straw. Products with specific surface area above 2000 m²/g. The electrochemical tests showed that when modified by N, Mn and mixed with SiO ₂, the capacitance value was significantly improved, reaching 169.3 respectively; 199.7 and 181.7 F/g at a scan rate of 2 mV/s. Capacitance is still preserved after many charge-discharge cycles at varying scan potentials and amperages.

Recommendations

Through the results published in the thesis, we recommend to conduct a number of follow-up studies as follows:

1. Expand the research results of the project on pilot scale, in order to proceed to apply it in practice.

2. Expanding the scope of application of research processes towards utilizing carbon and silica from other waste biomass sources such as corn cobs, coffee husks, sugarcane baits, etc., as well as lignin- and alkaline-rich black waste from factories biofilters and paper production.

NEW CONTRIBUTIONS OF THE THESIS

1. Introduce the process of preparing biochar modified by different salts by hydrothermal carbonization method, which is a green method that limits the use of chemicals, heat and makes use of agricultural waste. This is the first work in Vietnam to synthesize biochar with high adsorption capacity from straw by single-stage hydrothermal method.

2. Producing the process of preparing magnetic biochar by two-stage hydrothermal carbonization method. This is the first work to publish biochar material with high magnetic and adsorption capacity from lignin extract of straw.

3. Introduce the process of preparing SiO ₂ from rice husk ash by dissolution - precipitation method. The product has a crystalline structure and uniform particle size distribution, suitable for mixing with activated carbon used as electrode materials.

4. Presenting a process for preparing N and Mn modified activated carbon from lignin extract in KOH by pyrolysis in an inert gas environment, an economical and environmentally friendly method. Activated carbon products have high electrochemical performance that is superior to other biomass-derived materials.

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