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# Hydrochar Production Methods: Comparative Insights into Hydrothermal and Microwave Processes

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## ABSTRACT

This study compares hydrochar production from agricultural waste using conventional hydrothermal carbonization (HTC) and microwave-assisted carbonization methods. Wheat straw (HWS), rice straw (HRS), and bagasse (HBG) were used as feedstocks. Microwave-assisted carbonization resulted in higher yields and distinct chemical structures compared to conventional HTC. Microwave hydrochars (m-HWS, m-HRS, m-HBG) showed lower surface areas but increased pore volumes and thermal stability. They also exhibited enhanced heavy metal adsorption capacities, particularly at higher pH levels. These findings highlight the advantages of microwave-assisted carbonization for producing hydrochar with superior properties, offering insights for optimizing production processes and expanding applications.

**Keywords:** hydrochar; biochar; hydrothermal carbonization; biomass; agriculture waste

## Introduction

The increasing generation of agricultural and industrial waste has necessitated the development of effective and sustainable waste management strategies. Among these, the conversion of biomass into hydrochar via hydrothermal carbonization (HTC) and microwave-assisted methods presents a promising approach <sup>1-4</sup>. Hydrochar, a carbon-rich material, offers potential applications in energy production, soil amendment, and environmental remediation, particularly in the adsorption of pollutants from water and air. Conventional hydrothermal carbonization involves subjecting biomass to high temperatures and pressures in the presence of water, resulting in the formation of hydrochar with specific physicochemical properties. However, recent advancements have introduced microwave-assisted carbonization as an alternative

method, which promises higher efficiency and enhanced material properties through rapid heating and energy transfer<sup>5-7</sup>.

This study aims to systematically compare the properties and performance of hydrochar produced by conventional HTC and microwave-assisted methods. By focusing on three common biomass sources—wheat straw (HWS), rice straw (HRS), and bagasse (HBG)—this research seeks to elucidate the differences in yield, elemental composition, surface area, and functional groups between the two production techniques. Moreover, the study investigates the thermal stability and adsorption capacities of the resulting hydrochars, providing a comprehensive understanding of their potential applications.

Through detailed characterization and comparative analysis, this research contributes to the growing body of knowledge on biomass conversion technologies, highlighting the transformative effects of microwave irradiation on hydrochar properties. The findings of this study will inform future efforts to optimize hydrochar production processes, enhance material performance, and expand the practical applications of hydrochar in various environmental and industrial contexts.

## **Methodology**

For Hydrothermal carbonization via conventional oven Agricultural residues, including rice straw, wheat straw, and bagasse, were obtained from local field and farms in India for the synthesis of hydrochar. Approximately 60 grams of naturally dried agricultural waste separately, were combined with 400 ml of deionized (DI) water. This mixture was then introduced into a 500 ml stainless steel autoclave. The autoclave was subsequently subjected to heating, maintaining a temperature of 300 °C for a duration of 5 hours, while the pressure, monitored by a pressure gauge, was maintained at approximately 1000 psi. Following this controlled thermal treatment, the reactor was cooled to room temperature, allowing for the collection of the solid hydrochar product derived from the agricultural waste. Further, the agricultural waste hydrochar, was rinsed with DI water and was then dried at 80 °C in an oven. Once dried, the sample further processed through grinding and sieving to achieve a consistent particle size fraction ranging from 0.5 to 1.0 mm. To eliminate any remaining impurities, such as ash, rinsed by DI water, followed by another round of drying at 80 °C. The resulting hydrochar samples, now purified and free from contaminants, were stored for future experimental use. For the production of modified hydrochar, Approximately 3 grams of the prepared hydrochar samples were immersed in a solution consisting of 20 ml of 10% hydrogen peroxide (H<sub>2</sub>O<sub>2</sub>) for a duration of 2 hours at room temperature, maintained at 22 °C with a permissible deviation of ±0.5 °C. After the reaction, the modified hydrochar underwent thorough rinsing with DI water and was

subsequently dried at 80 °C. The resulting modified hydrochar samples were carefully stored for future experimentation and applications.

For MHTC, around 1 g of Agriculture waste was loaded into the glass vial and 5 mL of distilled water was added, corresponding to the water to biomass ratio same as the amount found in previous work. The optimization of reaction conditions was based on the type of raw biomass used. The use of seaweed rich in cellulose necessitated the optimization of conditions to enhance cellulose conversion to hydrochar. Cellulose is a complex carbohydrate that is difficult to break down. Therefore, high temperatures and high pressures are typically required to effectively convert cellulose to hydrochar and longer reaction times are also needed to ensure complete conversion<sup>8</sup>. The sealed glass vial was put into the microwave, stirring started and the reactor was heated up to the set temperature of 200 °C, corresponding to the temperature as found in previous work within 20 min. Thereafter, the HTC process was maintained at the reaction temperature for a certain reaction time. During the process, the pressure has been measured and recorded by the machine. After the reaction, the reactor was cooled down to 60 °C, and then the liquid and solid products were separated by vacuum filtration with F1001 grade filter paper, size 125 mm. The liquid products were stored in the refrigerator at 4 °C for further analysis, and the solid char was dried in a conventional heating oven at 105 °C for 3 h and stored at room temperature<sup>6</sup>. The hydrochar produced by microwave hydrothermal treatment was designated as MHC, and hydrochar produced by conventional heating (with a reaction time of 4 h) was designated as HC.

### **Characterisation of the Hydrochar prepared by MHTC and HTC**

The investigation into the chemical composition and physical attributes of hydrochars and their efficiency derived from Wheat Straw (HWS), Rice Straw (HRS), and Bagasse (HBG) unveils nuanced insights into their potential applications across various domains Table 1. The hydrochars prepared by microwave assisted approach, likely subjected to various treatment protocols, exhibit distinct chemical compositions and physical properties comparing to classical hydrothermal technology. Beginning with m-HWS, it demonstrates a significant carbon content (C %) of 85.4% alongside a hydrogen content (H %) of 51.88%, indicating a substantial alteration from its raw form. Moreover, m-HWS displays an oxygen content (O %) of 5.89% and a nitrogen content (N %) of 38.22%, illustrating potential modifications aimed at enhancing nitrogen incorporation. The resultant O/C ratio of 0.22 and H/C ratio of 0.74 suggest a composition skewed towards carbon and hydrogen, with notable nitrogen enrichment. Additionally, m-HWS exhibits a specific surface area (BET) of 11.76 m<sup>2</sup>/g, implying a reduction from its untreated counterpart, possibly due to structural alterations induced by modification processes.

Moving to m-HRS, it presents a similar trend in composition modifications, featuring a carbon content of 82.56%, hydrogen content of 48.34%, oxygen content of 6.32%, and nitrogen content of 39.25%. These values reflect alterations intended to enhance nitrogen incorporation, with the resultant O/C ratio of 0.38 and H/C ratio of 0.81 indicative of modifications influencing the relative proportions of carbon, hydrogen, and oxygen. The specific surface area of m-HRS is measured at 6.89 m<sup>2</sup>/g, suggesting a reduction from its untreated counterpart, likely attributable to structural changes induced by modification treatments. Similarly, m-HBG showcases considerable modifications, characterized by a carbon content of 89.6%, hydrogen content of 60.22%, oxygen content of 6.82%, and nitrogen content of 38.78%. These modifications result in an elevated O/C ratio of 0.66 and a reduced specific surface area of 3.22 m<sup>2</sup>/g, suggesting substantial alterations in chemical composition and physical properties aimed at enhancing specific functionalities or applications.

**Table.1 Physio-chemical properties oh Hydrochars prepared by conventional and microwave assisted method**

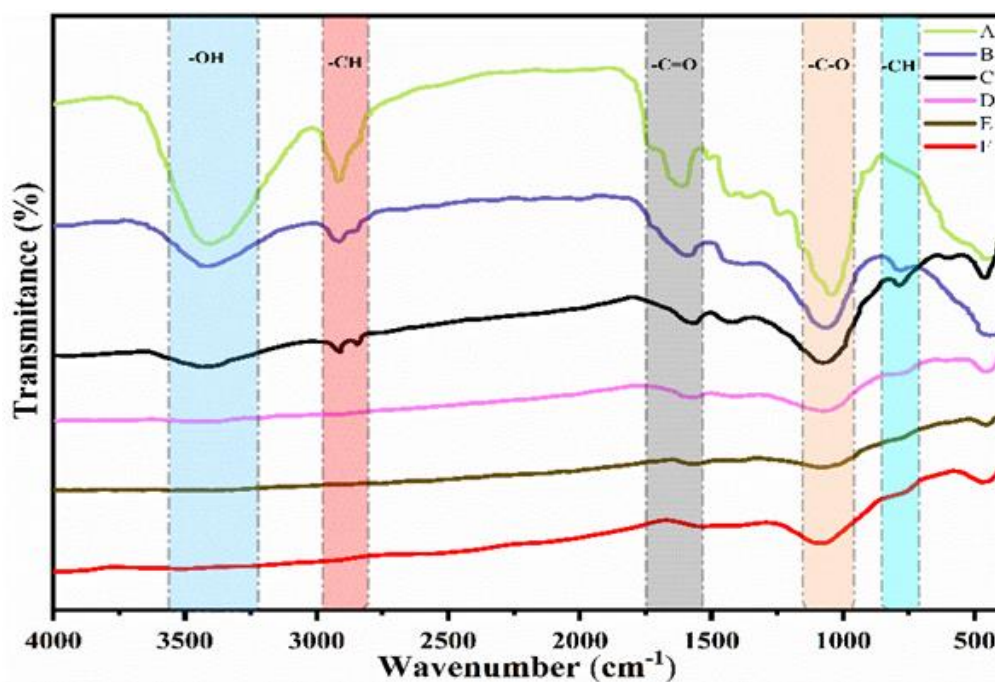
Hydrochar		Yield (%)	C (%)	H (%)	O (%)	N (%)	O/C	H/C	BET (m <sup>2</sup> /g)
Hydrochar	HWS	55.26	55.68	6.94	32.14	0.52	0.58	0.12	36.91
	HRS	48.72	60.74	4.92	31.62	0.22	0.52	0.08	38.42
	HBG	49.62	58.34	4.52	29.62	1.95	0.51	0.08	17.45
Microwave Hydrochar	m-HWS	85.4	51.88	5.89	38.22	0.22	0.74	0.11	11.76
	m-HRS	82.56	48.34	6.32	39.25	0.38	0.81	0.13	6.89
	m-HBG	89.6	60.22	6.82	38.78	0.66	0.64	0.11	3.22

### FTIR Analysis

Fourier Transform Infrared Spectroscopy (FTIR) is a powerful analytical technique widely employed in the characterization of hydrochars, offering invaluable insights into their chemical composition and structural properties. FTIR operates by measuring the absorption of infrared radiation by molecular vibrations within a sample, providing detailed information about functional groups present in the material. In the context of hydrochar characterization, FTIR facilitates the identification of key functional groups such as hydroxyl (OH), carbonyl (C=O), and carboxyl (COOH) groups, which play crucial roles in determining the material's reactivity and adsorption capacity. By analyzing FTIR spectra, researchers can discern changes in functional

group composition resulting from various production methods or modification treatments, aiding in the optimization of hydrochar synthesis processes. Moreover, FTIR enables the elucidation of structural transformations occurring during hydrothermal carbonization or modification processes, offering valuable insights into the mechanisms underlying hydrochar formation and functionality<sup>9,10</sup>. Overall, FTIR serves as an indispensable tool in the comprehensive characterization of hydrochars, facilitating a deeper understanding of their chemical composition, structural properties, and potential applications in fields ranging from environmental remediation to sustainable resource management.

FTIR analysis was conducted to investigate the surface functional groups of Hydrochar prepared using both the Hydrothermal Carbonization (HTC) method and microwave-assisted methods at varying power levels. The results, illustrated in Fig. 1, revealed notable differences in the detected functional groups between the two preparation methods. Specifically, Hydrochar prepared via microwave-assisted methods (mHWS, mHRS, and mHBG) exhibited fewer surface functional groups compared to those prepared using the HTC method (HWS, HRS, and HBG). For instance, peaks corresponding to aliphatic or aromatic  $\text{-OH}$  stretching ( $3421\text{ cm}^{-1}$ ) and aliphatic  $\text{C-H}$  antisymmetric stretching ( $2919/2850\text{ cm}^{-1}$ ) gradually diminished in intensity for HWS, HRS, and HBG, ultimately disappearing entirely in mHWS, mHRS, and mHBG, indicative of the decomposition and dehydration of aliphatic groups. This observation aligns with previous findings and suggests a transformative effect of microwave irradiation on hydrochar surface chemistry. Additionally, the peaks at  $1625\text{ cm}^{-1}$  (associated with carboxyl or carbonyl  $\text{C=O}$ ) and  $1061\text{ cm}^{-1}$  (linked to  $\text{C-O}$ ) weakened with increasing microwave power, indicating the decomposition of oxygen-containing functional groups—a trend consistent with the total acidity measurements of Hydrochar (Table 1). Interestingly, the peak at  $787\text{ cm}^{-1}$ , derived from bending vibrations of aromatic  $\text{C-H}$ , exhibited an increase in intensity with rising microwave power, suggesting an augmentation in the aromaticity of Hydrochar under microwave-assisted conditions. These findings underscore the nuanced effects of preparation methods and microwave power levels on the surface chemistry and functional group composition of Hydrochar, elucidating crucial insights for its application in various fields, including environmental remediation and resource management.



**Fig.1 FTIR analysis of Hydrochars prepared by both conventional and microwave assisted methods**

\*A= HWS (Hydrochar Wheat Straw) B= HRS (Hydrochar Rice Straw) C= HBG (Hydrochar Bagasse) D= m-HWS (Microwave Hydrochar WS) E=m-HRS (Microwave Hydrochar RS) F= m-HBG (Microwave Hydrochar BG)

### BET Analysis

The Specific Surface Area (SSA) and Pore Volume (PV) increased with the process selection from Hydrothermal Carbonization to Microwave assisted method for the preparation of Hydrochar (Table 1). This might be caused by an increase in the volatile organic component loss, leading to the formation of porous structure in the case of Microwave assisted method comparing to Hydrothermal Carbonisation method<sup>11,12</sup>. The results showed that mHBG had the highest SSA (156.09 m<sup>2</sup> g<sup>-1</sup>), which was 60.5 times higher than the value found for HWS. On the other hand, the PV of mHRS (0.0790 cm<sup>3</sup> g<sup>-1</sup>) was marginally higher than the 0.0741 cm<sup>3</sup> g<sup>-1</sup> of mHBG. This could be because, according to the Hydrochar skeleton collapsed at high pyrolysis temperatures, which corresponds to mHBG in the current investigation. The maximum hydrochar yield of 74.66% was achieved with HWS, while hydrochar yields fell as microwave power increased. This was due to the biomass heating rate being quicker and the greater output of bio-oil and syngas under high power<sup>13</sup>. The oxygen-containing functional groups are main source of surface acidity of carbon materials<sup>14</sup>, moreover play an important role for metal ion binding with Hydrochar thus influencing adsorption ability. It could also be seen in Table 1 that there is significant decrease in the total acidity in Microwave assisted hydrochars, this was because oxygen-containing functional groups are easily decomposed at high temperatures.

Surface area may supply the sites for adsorption processes in carbon adsorbents, hence a larger SSA often translates into better adsorption performance<sup>15</sup>. The SSA of microwave hydrochar in the current investigation ranged from 2.6 to 156.1 m<sup>2</sup> g<sup>-1</sup>, which was significantly greater than in other investigations that employed microwave pyrolysis without the addition of an activated carbon catalyst. For instance, the SSA of Hydrochar from mHWS, mHRS, and mHBG ranged from 3.9 to 4.9 m<sup>2</sup> g<sup>-1</sup>. Similarly, the SSA of WS hydrochar that was microwave-pyrolyzed was less than 4.5 m<sup>2</sup>g<sup>-1</sup>. The advantages of activated carbon catalyst on the pyrolysis process and the characteristics of hydrochar were validated by these comparisons.

### **Thermo-gravimetric Analysis (TGA)**

Thermo-gravimetric Analysis (TGA) serves as a vital tool in the characterization of hydrochar, offering valuable insights into its thermal stability, decomposition behavior, and composition. In light of the provided inputs, TGA plays a pivotal role in elucidating the impact of different synthesis methods and conditions on the thermal properties of hydrochar. The Thermo-gravimetric Analysis (TGA) curves depicted in Fig. provide valuable insights into the thermal stability and recalcitrance of hydrochar derived from various agricultural waste sources. These curves, which illustrate the change in mass of the hydrochar samples as a function of temperature, reveal distinct patterns influenced by the preparation method and biomass used. Notably, the weight loss of biochar is notably affected by microwave power, showing a decrease with varying agriculture biomass. Analyzing the TGA curves allows for the identification of three distinct stages of decomposition. The initial stage is characterized by a slight weight loss, attributed primarily to the evaporation of volatile components and moisture present in the Hydrochar. Remarkably, the end temperatures of this stage exhibit a notable increase, ranging from 190.17 °C for HWS to 296.16 °C for m-HBG, suggesting variations in the thermal properties of hydrochar derived from different agricultural waste sources.

The most significant weight loss occurs during the second stage, attributed to the volatilization of free and nonstructural water, as well as functional groups present in the hydrochar. The extent of weight loss during this stage varies among the different hydrochar samples, with values ranging from 44.89% to 64.98%, highlighting differences in composition and thermal behavior. In the final stage, the rate of weight loss decreases gradually with increasing temperature, indicating a higher degree of carbonization and improved thermal stability of biochar prepared under higher microwave power conditions. Additionally, the decreased atomic ratios of hydrogen to carbon (H/C) and oxygen to carbon (O/C) further corroborate the increase in aromaticity and stability observed with the transition from hydrothermal carbonization to microwave power-assisted methods. These findings underscore the importance of TGA analysis in elucidating the

thermal properties and decomposition behavior of hydrochar, providing valuable insights for optimizing synthesis protocols and tailoring hydrochar properties for specific applications in environmental and industrial settings.

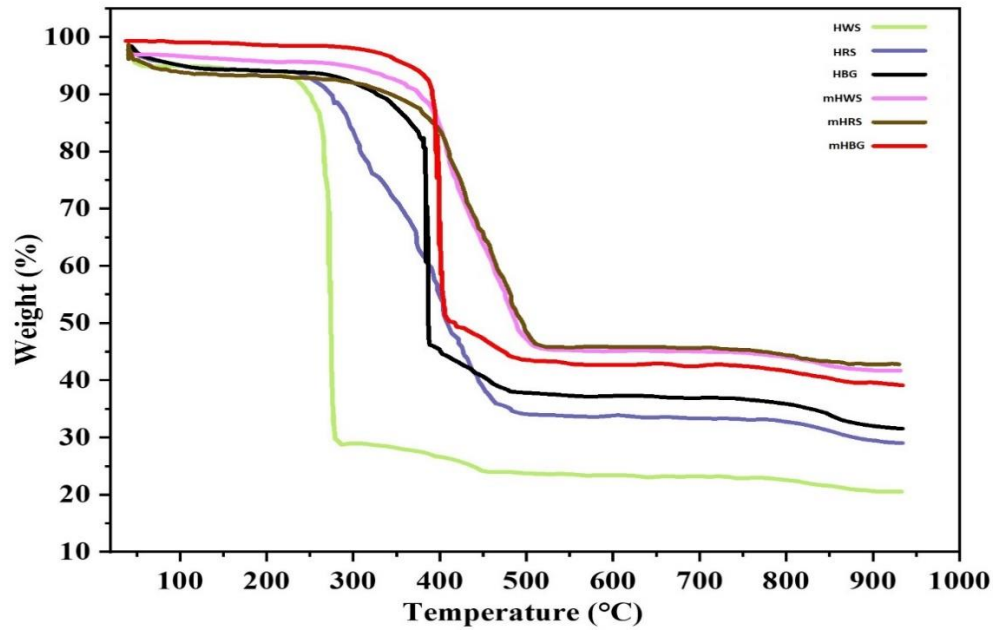
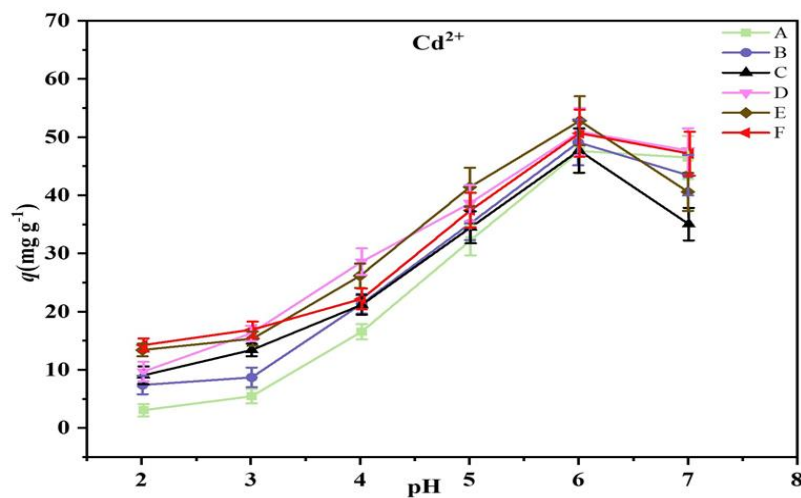


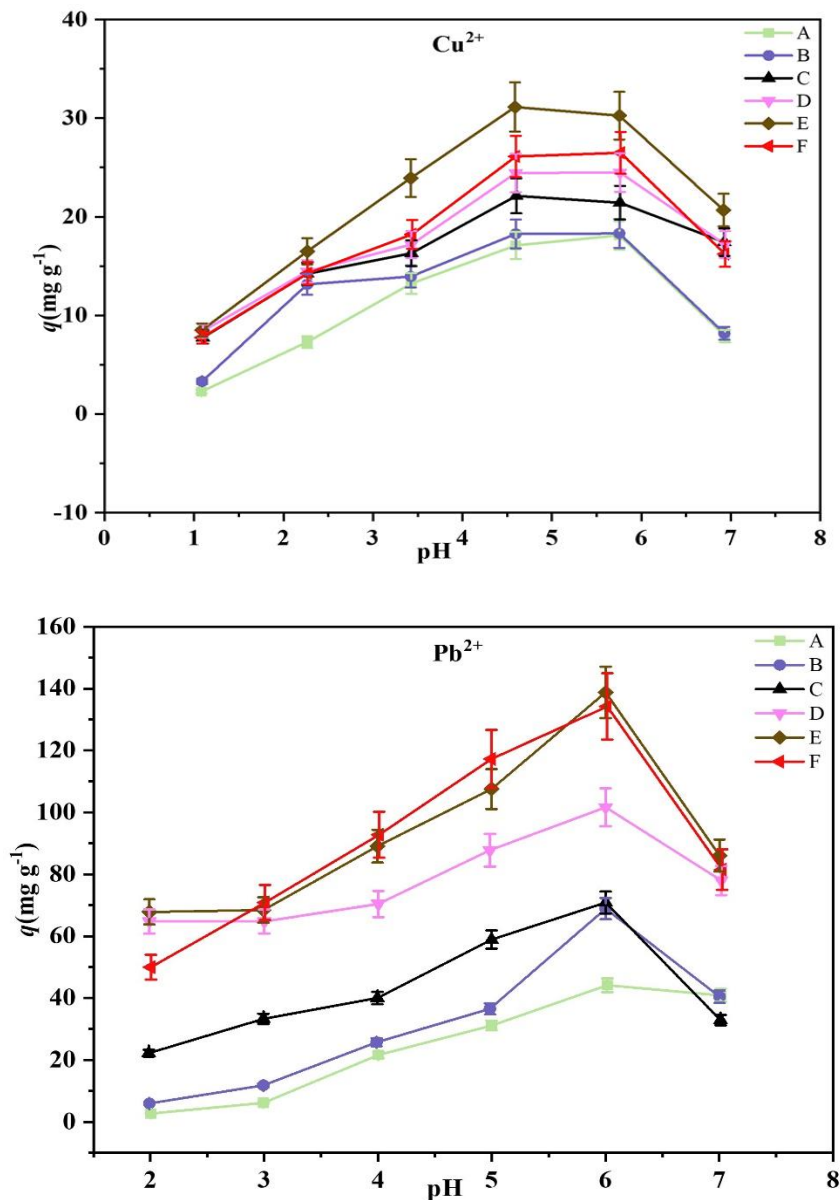
Fig 2. TGA analysis of Hydrochar produced by conventional method and Microwave assisted method

### Adsorption of heavy metals

The pH of the solution is often strongly connected to the surface charge of the adsorbent and the speciation of metal ions in the aqueous solution <sup>16,17</sup>. The **Fig. 3** displays the heavy metal adsorption capabilities on several types of Hydrochar in the initial solution's pH range of 2–7.







**Fig.3 Adsorption capacity of Heavy metal with varying pH**

\*A= HWS (Hydrochar Wheat Straw) B= HRS (Hydrochar Rice Straw) C= HBG (Hydrochar Bagasse) D= m-HWS (Microwave Hydrochar WS) E=m-HRS (Microwave Hydrochar RS) F= m-HBG (Microwave Hydrochar BG)

Hydrochar made from various agricultural wastes had a similar tendency of adsorbing Pb<sup>2+</sup>, Cd<sup>2+</sup>, and Cu<sup>2+</sup> as pH increased. The adsorption capabilities of heavy metals decreased with decreasing pH. The explanation most likely suggested that metal ions and plentiful H<sup>+</sup> compete for adsorption sites on the surface of hydrochar<sup>18,19</sup>. The explanation most likely suggested that metal ions and plentiful H<sup>+</sup> compete for adsorption sites on the surface of hydrochar. However, the majority of the functional groups on the hydrochar surface which are essential for the adsorption of heavy metals—were protonated by H<sup>+</sup> in the solution. Consequently, an electrostatic repulsion resulted from the adsorbent and adsorbate being simultaneously

positively charged. The adsorption capacity of biochar increased significantly as pH increased. For  $Pb^{2+}$ ,  $Cd^{2+}$ , and  $Cu^{2+}$ , the maximum adsorption capacities were reached at pH increases of 6 and 5, respectively, at which point the adsorption quantities were  $Pb^{2+}$  with 139.44 mg g<sup>-1</sup>,  $Cd^{2+}$  with 52.92 mg g<sup>-1</sup>, and  $Cu^{2+}$  with 31.25 mg g<sup>-1</sup>. Previous researches have revealed similar results about the maximum adsorption capabilities for different plant-based hydrochars. Increased pH resulted in the loss of protons by oxygen-containing functional groups, a decrease in the positive charge on the surface of adsorbents, a weakening of the competition between protons and heavy metal ions on adsorption sites, and the release of adsorption sites, which increased adsorption capacity. The adsorbing capabilities of  $Cd^{2+}$ ,  $Pb^{2+}$  and  $Cu^{2+}$  declined during a further pH rise to 7, which was partly due to metal ions starting to precipitate as hydroxides. Since  $Pb(OH)_2$  and  $Cd(OH)_2$  were found to begin precipitating at pH 5.5 and 6.5, respectively, the starting pH of the solution in this investigation was lower than 7. When comparing the adsorption capacities of biochar prepared under different microwave powers, it was noted that mHRS showed higher adsorption performance for heavy metals even than mHBG.

## Conclusion

This study provides a comprehensive comparison of conventional hydrochar and microwave hydrochar derived from various agricultural wastes, specifically wheat straw (HWS), rice straw (HRS), and bagasse (HBG). Microwave hydrochar generally demonstrated higher yields, enhanced elemental oxygen content, and higher atomic O/C and H/C ratios compared to conventional hydrochar. Despite a lower BET surface area in microwave hydrochar, the structural and compositional alterations brought by microwave-assisted methods have significant implications for their surface chemistry and functional group composition. FTIR analysis highlighted that microwave-assisted hydrochars exhibit fewer surface functional groups, indicating significant decomposition and dehydration under microwave conditions. Additionally, microwave hydrochar exhibited higher specific surface area (SSA) and pore volume (PV) due to the formation of a more porous structure, attributed to the loss of volatile organic components. Thermal Gravimetric Analysis (TGA) revealed distinct stages of decomposition, with microwave hydrochars showing improved thermal stability and higher degrees of carbonization. The study also demonstrated the impact of solution pH on the adsorption capacities of heavy metals by hydrochars, with microwave hydrochar showing enhanced adsorption performance at higher pH levels.

## Future Recommendations

Future research should explore the optimization of microwave power and process parameters to maximize the beneficial properties of hydrochar, such as yield, specific surface area, and adsorption capacity. Investigating the use of different catalysts during microwave-assisted hydrochar production could further enhance the material properties and performance. Additionally, a more detailed analysis of the mechanisms underlying the decomposition of surface functional groups and the formation of porous structures in microwave hydrochar would provide valuable insights for tailoring hydrochar for specific applications. Further studies should also examine the long-term stability and reusability of microwave hydrochar in environmental applications, particularly in the adsorption of various contaminants. Finally, expanding the range of biomass sources and examining their effects on the properties and performance of microwave hydrochar would contribute to the development of more versatile and efficient carbon materials.

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