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Characterization and use of hydrochars from wheat straw, fruit peels, and sewage sludge: A potential biofuel source

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ABSTRACT: This study explores the hydrothermal carbonization (HTC) of wheat straw (WS), fruit peels and pulps (PP), and solid sewage sludge (SS) to produce hydrochar with enhanced fuel properties. Samples were collected, dried, ground, and sieved, then subjected to HTC at temperatures of 180°C, 220°C, and 260°C for one hour. The hydrochars were analyzed for solid yield, high heating value (HHV), energy densification, fuel ratio, and surface morphology. Results indicated a decrease in solid yield with increasing temperature of all the hydrochars. WS and PP hydrochars showing increased HHV and energy densification with temperatures increase. SEM analysis revealed significant structural changes, including increased surface macropores and roughness, enhancing combustion properties. WS-260 and PP-260 hydrochars exhibited the highest potential as biofuels due to their high HHV, energy yield, and favorable morphological characteristics. Conversely, SS-derived hydrochars showed lower HHV but increased homogeneity and particle fragmentation. The findings suggest that HTC-treated WS and PP, particularly at 260°C, can serve as sustainable and efficient solid biofuels, offering a viable alternative to traditional fossil fuels and contributing to effective waste management.

Key words: Fruit peels and pulps, hydrochar, Hydrothermal carbonization, sewage sludge, solid biofuel, wheat straw

In recent decades, global waste generation has significantly increased, exceeding two billion metric tons of municipal solid waste (MSW) annually. Projections indicate a potential rise of approximately 70 percent by 2050 (Alves, 2023). Additionally, the annual generation of agricultural waste is 140 billion tons worldwide, primarily consisting of residues and byproducts of agricultural activities such as straws (Maji *et al.,* 2020). Rapid economic growth, improved living standards, urbanization, and industrialization have substantially contributed to this increase in global solid waste production (Chen *et al.,* 2021). Biowaste, a major contributor to this scenario, is expected to see a substantial annual increase, reaching a global generation of 100 billion metric tons (Cho *et al.,* 2020). This growth in biowaste is closely tied to population expansion, occupying significant space and causing contamination of soil, groundwater, and air. Furthermore, the presence of toxic compounds in biowaste poses potential risks to human health through various biological pathways. Beyond being a significant environmental pollutant, biowaste represents a substantial reservoir of biomass resources containing bioavailable organic materials (Guo *et al.,* 2022). Originating primarily from industrial and agricultural activities, municipal engineering, and daily life, biowaste is both biodegradable and transformable.

Various techniques, including biochemical, physicochemical, and thermochemical processes, can transform renewable biomass sources into solid, liquid, and gaseous products. Thermochemical processes, such as pyrolysis and gasification, convert organic waste into solid carbonaceous output (Sharma *et al.,* 2019). These methods avoid the need for microorganisms, allowing completion within minutes to hours (Yeoh *et al.,* 2018). Slow pyrolysis, a thermochemical technique, produces biochar, bio-oil, and syngas, offering versatile biofuel applications like energy storage, purification, and soil enhancement (Karakas *et al.,* 2017). However, dealing with raw biomass with high moisture content (>60%) poses economic challenges, requiring larger storage and controlled drying processes (Elkhalifa

et al., 2019).

The wet waste conversion method known as hydrothermal carbonization (HTC) has gained recent attention and thorough investigation (Kambo and Dutta 2015; Saqib *et al.,* 2019). HTC utilizes a series of reactions, including hydrolysis, dehydration, decarboxylation, aromatization, condensation, and polymerization, to leverage the high moisture content of waste, transforming it into a carbon-rich substance called hydrochar (HC) (Hoekman *et al.,* 2011). Typically conducted in a sealed vessel with excess water (over 70% by weight) and temperatures ranging from 180 to 260°C, HTC produces two main products: process wastewater and hydrochar, a solid material enriched with carbon. Due to its exceptional adaptability to wet waste, HTC has been extensively studied with various high-moisture feedstocks, including MSW, specific lignocellulosic biomass like paper mill sludge, microalgae, fruit waste and peels, as well as non-lignocellulosic waste (Zhai *et al.,* 2016; Chen *et al.,* 2017). Significantly, the HTC process produces hydrochar that is easily grindable, has higher energy density, and exhibits hydrophobic properties, taking advantage of the presence of water (Kambo and Dutta, 2015). Under subcritical heat conditions, the inherent moisture in wet biodegradable waste serves as an organic solvent and catalyst. This environment, known for preserving the stability of aromatic compounds, makes hydrothermal pre-treatment particularly advantageous for enhancing the aromaticity of solids (Funke and Ziegler, 2010).

The use of hydrochar offers a promising solution for managing biomass residues, especially those with high moisture content, reaching up to 80%. Extensive efforts have focused on producing energy-rich hydrochars from diverse sources like digestate, lignocellulosic biomass, and manure. The quality of the resulting hydrochar depends on various HTC operational conditions, including feedstock type, temperature, and duration (Saqib *et al.,* 2019). Hydrochar shares structural similarities with coal, distinguishing it from biochar produced through dry pyrolysis. However, a Comprehensive characterization of hydrochar from various biowastes is needed to identify the most suitable and efficient types for fuel applications.

Keeping this in view, the present study was undertaken with the following objectives: 1. To explore the HTC of agricultural, industrial, and municipal solid wastes across varying thermal conditions with a constant retention time. 2. To identify the potential of hydrochar as fuel by comprehensively analyzing the yield, fuel properties, and morphological attributes of the hydrochar produced from the biosolid wastes.

MATERIALS AND METHODS

Sample collection and preparation

Wheat straw (WS) from the UP 2938 wheat variety was collected immediately after threshing at the Crop Research Centre of G.B. Pant University of Agriculture and Technology in Pantnagar, India. Solid sewage sludge (SS) was obtained from a common effluent treatment plant in Rudrapur using a polyethylene pail. Fruit peels and pulps (PP) were sourced from Kanak Food Processing Industry in Haldwani, India. Representative samples of WS, SS, and PP were collected using the quartering method. The samples underwent drying, crushing, and sieving for preparation. They were air-dried at ambient temperature for three days to remove excess moisture. The dried samples were finely ground using a 'Heavy Duty' Willy mill powered by a 1 H.P. electric motor operating on 220 Volts. The ground samples were then sieved through a No. 18 mesh (1 mm), with particles smaller than 1 mm collected. These sieved samples were stored in zip-lock bags for further research and analysis.

Hydrochar preparation

The HTC process for the prepared WS, PP, and SS was conducted in a 150 mL Teflon-lined hydrothermal autoclave (Figure 1). This autoclave featured an outer layer made of high-quality stainless steel (SS-316) and an inner lining of Polytetrafluoroethylene (PTFE), also known as Teflon, (Om Tech Solutions, Rudrapur, India). The experimental conditions were determined based on a study by Cai *et al.* (2016). For each experiment, 8

g of the prepared WS, SS, and PP samples were individually mixed with 100 mL of deionized water. The study aimed to understand the effects of heating at different temperatures (180°C, 220°C, and 260°C) for a constant residence time of 1 hour on the structural, functional, and morphological properties of the hydrochars. The heating time refers to the duration required for the reactor to reach the desired temperature. Once the desired temperature was achieved, the reactor was maintained at that temperature for the specified reaction time.

After placing the prepared sample in the HTC autoclave, it was sealed and placed in an electric furnace, then gradually heated to the desired temperature at a rate of 15 ± 5°C per minute. After the specified reaction time, the autoclave was removed from the oven and transferred to a cold-water bath. It is important to note that the cooling phase, lasting approximately 20-25 minutes, was not included in the overall reaction time. Following cooling, the solid and liquid phases were separated by vacuum filtration. The resulting dark brown powder, referred to as "hydrochar (HC)," was dried in an oven at 105°C for 24 hours and stored in airtight vials for further analysis.

The nomenclature for the hydrochars were determined by the sample source and the reaction temperature. For example, hydrochar prepared from wheat straw at 180°C was labeled as "WS-180." Similarly, other hydrochars were named following the same pattern: WS-220, WS-260, SS-180, SS-220, SS-260, PP-180, PP-220, and PP-260, according to the specific treatment conditions.

Fuel characterization methods

The experimental high heating values (HHV) were measured by an oxygen bomb calorimeter (BC) from Parr (Model 1341 Plain Jacket Calorimeter) according to the UNE-EN ISO9831:2004 standard method. Moisture content (MC), ash content (AC), and volatile matter (VM) content, was carried out following the protocols established by ASTM International Standards: ASTM 871-82, 2006, ASTM-D 3174-04, 2009, and ASTM-D 3175-07. The fixed carbon (FC) content was calculated by subtracting the MC, AC, and VM content from 100% on a dry basis. Solid yield, energy yield, fuel ratio, and energy densification for all raw and hydrochar samples were calculated based on the following formulas:

Solid yield (%)	(Hydrochar weight) / (Raw sample weight) × 100	Sharma and Dubey (2020)
Fuel ratio	Fixed Carbon / Volatile Matter	
Energy densification	HHV of hydrochar / HHV of raw sample	
Energy yield (%)	Hydrochar solid yield × Energy densification	

Surface morphology characterization

The surface morphology of the hydrochar samples was examined using a JEOL JSM-6610LV Scanning Electron Microscope (SEM). Samples were first cleaned with distilled water and ethanol, then dried at 60°C for 24 hours. After mounting on aluminum stubs with carbon adhesive tape, the samples were sputter-coated with a 10 nm layer of gold. SEM imaging was conducted in low vacuum mode at an accelerating voltage of 15 kV and a working distance of 10 mm. Images were taken at magnifications ranging from 100x to 5000x to analyze surface features. The figures were generated using OriginPro, Version 2024. OriginLab Corporation, Northampton, MA, USA.

RESULTS AND DISCUSSION

Fuel characterization

Table 1 furnishes details on solid yield, HHV, fuel ratio, energy yield, and Energy densification for all raw and hydrochar samples. The solid yield of hydrochar samples from WS, PP, and SS demonstrated a decreasing trend with increasing HTC temperature. This decline is attributed to the generation of liquid and gaseous by-products and the extraction of cellulose and hemicellulose components (Kambo and Dutta, 2015). The highest solid yield was recorded at an HTC temperature of 180°C, while the lowest yield was observed at 260°C. These findings suggest that the predominant

Table 1: Solid yield, higher heating value (HHV), fuel ratio, energy yield (EY), and energy densification (ED) for all the raw and hydrochar samples. ***(Note: WS-Wheat straw, PP-Fruit peels and pulps, and SS-Sewage sludge)***

Samples	Solid Yield (%)	HHV (MJ/kg)	Fuel ratio	ED	EY (%)
WS	-	14.53	0.04	-	-
WS-180	69.5	16.80	0.13	1.16	80.34
WS-220	59.8	21.22	0.19	1.46	87.36
WS-260	48	26.59	0.36	1.83	87.84
PP	-	15.04	0.20	-	-
PP-180	40	19.70	0.29	1.31	52.40
PP-220	32	23.63	0.36	1.57	50.26
PP-260	31	26.32	0.42	1.75	54.25
SS	-	14.29	0.08	-	-
SS-180	70.2	12.55	0.31	0.88	61.68
SS-220	62.5	10.69	0.41	0.75	46.78
SS-260	58	10.00	0.47	0.70	40.60

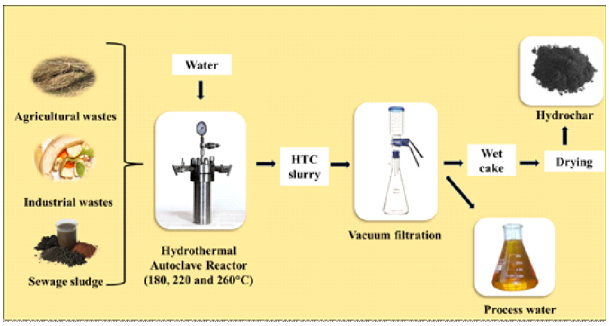

Fig. 1: Schematic diagram of hydrochar production from various biowastes through HTC process in this research

factor influencing hydrochar yield is the reaction temperature(Wang *et al.,* 2020). The HHV of hydrochars derived from WS and PP exhibited an increase with the elevation of HTC temperature. The maximum Carbon (C) content was identified in WS-260, resulting in the highest HHV value recorded at 26.59 MJ/kg. HHV in hydrochars is directly correlated with C content and inversely correlated with Oxygen (O) content. As the intensity of the HTC process rises, reactions such as decarboxylation become more prominent, leading to a higher C content and a lower O content (Basso *et al.,* 2015). In contrast, the HHV of hydrochars derived from SS was lower than that of raw SS, possibly due to a high AC and lower C content (Wang *et al.,* 2020). The utilization of SS-derived hydrochar for fuel applications may face economic challenges due to potential issues of slagging and fouling during char combustion, stemming from the high AC.

The energy densification value increased after HTC for hydrochars derived from WS and PP (greater than 1 for all sample types). The enhancement in the Energy densification value is ascribed to a reduction in solid mass caused by dehydration and decarboxylation reactions during HTC, resulting in increased energy densification (Berge *et al.,* 2011). However, the Energy densification value of hydrochar derived from SS was less than 1 due to its low HHV. The energy yield of hydrochars produced from WS, PP, and SS followed a similar trend to that of the energy densification. All hydrochars reported an increasing fuel ratio with increasing HTC temperature, as the FC content increased with the HTC temperature. The highest and lowest fuel ratios were calculated among hydrochars, with PP-260 having the highest and WS-180 having the lowest. A higher fuel ratio indicates improved stability of the hydrochar, allowing for higher firing temperatures, greater flame stability (less violent flame), and reduced heat loss during combustion (Mannarino *et al.,* 2022).

Surface morphological characterization

The SEM images in Figure 2 depict the surface morphological characteristics of both the raw samples and their corresponding hydrochars. The surface of the original WS appeared smooth, with a dense fiber structure. As the HTC temperature increased, there was a noticeable escalation in the damage to the fiber structure. Precisely, at an HTC temperature of 180 °C, the fiber structure of WS

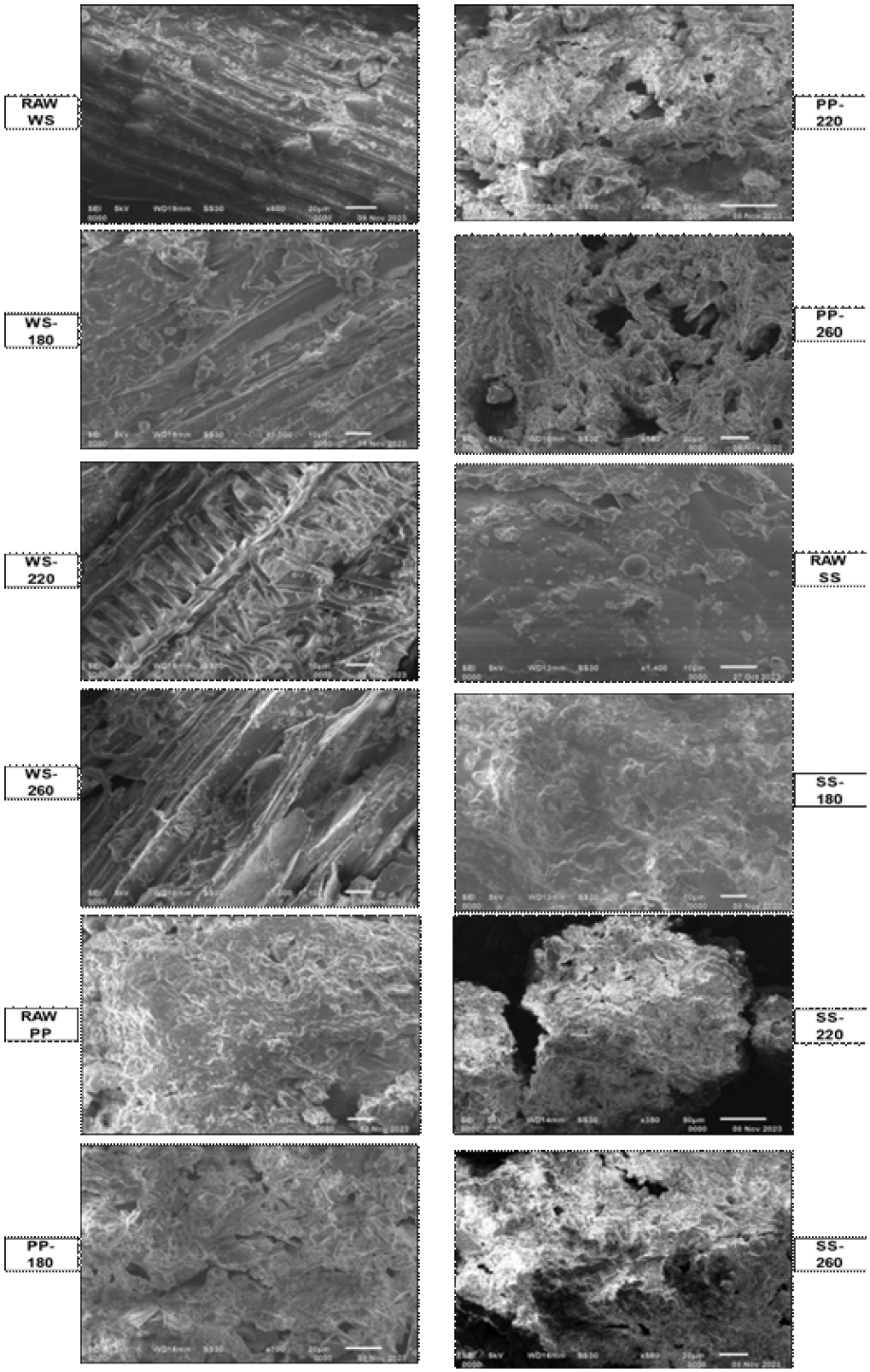

Fig. 2: Scanning Electron Microscope images for raw samples and its hydrochars

experienced substantial disruption. This was attributed to the breakdown of hemicellulose, cellulose depolymerization, and softening of lignin (Zhang *et al.,* 2020). Upon elevating the HTC temperature, surface deformation was observed in WS-220 and WS-260. This deformation can be attributed to the thermal degradation of lignin, a process initiated above 200 °C during HTC. Notably, the HTC treatment at 260 °C led to crack formation on the WS surface, introducing a feature that could potentially enhance the versatility of this hydrochar for various applications.

The SEM images reveal a distinct transformation in the structure of the samples corresponding to an increase in HTC temperature. Initially, the raw samples exhibit a well-defined structure characterized by intact textures. However, after HTC treatment, the smooth surfaces of the raw PP underwent a transition to rough textures, a consequence of the decomposition of cell walls (Pala *et al.,* 2014). In the case of PP-260 hydrochar, complete degradation occurred, leading to the breakdown of large polysaccharide molecules into disaccharides and monosaccharides, eventually atomizing into carbonaceous particles Significantly, even at an HTC treatment temperature of 180 °C, considerable degradation similar to the PP surface was evident (Xiao *et al.,* 2012). This suggests that thermal decomposition can initiate at temperatures lower than 200 °C. Hydrochar produced from PP treated at 220 °C and 260 °C exhibited a distinct morphology, characterized by spherical particles with a uniform size distribution (Gai *et al.,* 2016).
The surface characteristics and morphology of raw SS underwent consistent alterations with increasing HTC temperature. Elevated reaction severity resulted in SS-derived hydrochars displaying increased homogeneity and the formation of small particles due to fragmentation (Falco *et al.,* 2011). This transformation indicated the disruption of flocs and cellular tissues in SS, a consequence of gas volatilization and chemical bond scission during the HTC process. The overall porosity of raw SS was augmented by the HTC process, attributed to dehydration and the release of volatile substances (Puccini *et al.,* 2019). Notably, at high reaction temperatures (SS-260), the presence of sphere-like aggregates was observed. This observation suggests that the aromatization and polymerization processes in HTC might require higher reaction temperatures. It is hypothesized that these sphere-like aggregates, consisting of a hydrophobic aromatic structure and a hydrophilic shell, could be associated with the HTC reaction of polysaccharides in organic sludge (Sevilla and Fuertes, 2009).

Selection of potential hydrochar for fuel application

Hydrochar produced from WS at 260°C (WS-260) demonstrated a higher HHV, energy yield, and energy densification compared to other samples. SEM images revealed that WS-260 also had a significant amount of surface macropores, which can enhance its combustion properties. On the other hand, hydrochar produced from PP at 260°C (PP-260) exhibited a higher fuel ratio and substantial surface pore space. The lower yield observed in these hydrochars is attributed to the generation of liquid and gaseous by-products and the extraction of cellulose and hemicellulose components, which reduces the pollutants emitted during combustion. These properties suggest that WS-260 and PP-260 hydrochars may have a structure similar to lignite, indicating their potential suitability as solid biofuels. The results highlight the promise of these hydrochars for sustainable energy applications, with their structural and compositional characteristics making them viable alternatives to traditional fossil fuels.

CONCLUSION

The study demonstrates that HTC temperature profoundly influences the solid yield, HHV, energy densification, fuel ratio, and surface morphology of the WS, PP, and SS hydrochars. Higher HTC temperatures generally enhance the carbon content and HHV, particularly in WS and PP, due to intensified decarboxylation and dehydration reactions. This leads to hydrochars with increased energy densities, making them more efficient for combustion. Surface morphological changes observed through SEM imaging further support these findings. The disruption and degradation of the fiber

structures in WS and PP at higher HTC temperatures result in the formation of macropores and increased surface roughness, enhancing their combustion properties. In contrast, SS-derived hydrochars, despite lower HHV, show increased homogeneity and particle fragmentation, indicative of significant structural transformations. Among the samples, WS-260 and PP-260 hydrochars exhibit the most promising characteristics for fuel applications, with high HHV, energy yield, and desirable morphological features. These hydrochars demonstrate potential as sustainable energy sources, offering an eco-friendly alternative to conventional fossil fuels and contributing to waste management solutions. Future research should prioritize the economic and environmental evaluation of hydrochar to ensure its sustainable application.

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