



## Microporous and Mesoporous Activated Carbons from Tea Stalk and Tea Stalk Pulps: Effect of Lignin Removal by One-Step and Two-Step Organosolv Treatment

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**Abstract:** Delignification is a crucial pretreatment in the production of diverse value-added products from lignocellulosics. While modifying the surface functional groups, delignification also increases the specific surface area by providing a porous structure to the lignocellulosic biomass. Hydrothermal pretreatment can be used prior to delignification, to recover hemicellulose and boost delignification. By removing lignin and hemicellulose, cellulose-rich pulp becomes more accessible for activation. In the present study, three different activated carbons were prepared: activated carbon from tea stalk itself (ATS), activated carbon from tea stalk pulp obtained by using glycerol organosolv pretreatment (ATP), activated carbon from tea stalk hydrochar pulp obtained by using sequential hydrothermal pretreatment-organosolv delignification (AHTP). Each precursor was carbonized (at 800 °C) in the presence of KOH (KOH/precursor: 2/1). Activated carbons were characterized for their elemental content, surface functional groups, thermal stability, crystallinity, surface morphology, surface area and porous structure using elemental analysis (C-H-N-S), FTIR, TGA, XRD, SEM and, BET analysis, respectively. While hydrothermal pretreatment prior to organosolv pulping reduced the delignification yield, it also altered the pore structure of activated carbon. Among the activated carbons, only ATS had microporous structure with an average pore radius of 1 nm. ATP had the highest surface area (2056.72 m<sup>2</sup>/g) and micropore volume (0.81 cm<sup>3</sup>/g). Having mesopores (with an average pore radius of 5.74 nm) in its structure, AHTP had the least micropore volume (0.464 cm<sup>3</sup>/g) and surface area (1179.71 m<sup>2</sup>/g). The presence of micro and mesopores broadens the potential applications of activated carbon ranging from environmental applications to energy storage.

**Keywords:** Activated carbon, alkaline-glycerol organosolv treatment, hydrothermal pretreatment.

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### 1. INTRODUCTION

Activated carbons are carbon-rich porous materials which are prepared from different resources such as coal, coke, peat and biomass. Having the highest share of lignocellulosic biomass, agricultural wastes and residues are sustainable raw materials to produce value-added products such as activated carbon (1-3). Several lignocellulosic feedstocks have been preferred as precursors such as pineapple leaf and coconut shell (4), kenaf and rapeseed (5), corn straw (6), oil palm

leaves (7), olive bagasse (8), cherry stones (9), almond shell wastes (10). Due to their high surface area, variable porosity and surface functional groups, activated carbons are used as adsorbents for the removal of water pollutants and air pollutants, as catalyst support for preparing catalyst, as electrodes in supercapacitors for storing ions, and as carbon nanotubes and carbon fiber.

The volume and diameter of pores, surface area, micropore volume and surface functional groups of

activated carbon are affected from the activating agent, activation type (physical or chemical), activation conditions, carbonization conditions, and the lignocellulosic precursor (11-13). The structural components of lignocellulose (namely cellulose, hemicellulose and lignin) have great impact on both yield and porous structure of activated carbon mainly due to their different chemical structure and thermal stability (14-17). In the study of Guo and Rockstraw (18), activated carbons of xylan, cellulose and lignin had different pore structures at same activation conditions. Lignin is the major component that contributes to the activated carbon yield, while cellulose, lignin and hemicellulose alter the porous structure (19, 20). Specific surface area and porosity of the activated carbon are higher as the precursor has high cellulose content (4). To take advantage of the high cellulose content, biomass can be partially delignified prior to activation/carbonization (6, 21-23). Chen et al. (6) fractionated cellulose, hemicellulose and lignin using NaOH and urea solution. Then, activated carbon samples were obtained by carbonizing those fractions under N<sub>2</sub> atmosphere at 800 °C in the presence of NaOH. Their study showed that activated carbon obtained from cellulosic fraction has better properties than activated carbon obtained from the lignocellulosic biomass itself. Mittal et al. (24) separated the cellulosic fraction by pretreating rice straw with an alkaline ionic liquid (choline hydroxide). The cellulosic fraction was carbonized at 600 °C for 3 hours under an argon atmosphere. In this study, adsorption capacity of mesoporous cellulose-based activated carbon was high. Han et al. (25) pre-treated poplar wood using CH<sub>3</sub>COOH and NaClO<sub>2</sub> solution at 80 °C for 18 hours. The cellulose-rich fraction was carbonized at 1000 °C for 2 h (under argon atmosphere). It was observed that the pore size of the pretreated activated carbon was lower than the activated carbon obtained from poplar wood. Sun and Hong (26) used different cellulose-based polymers ( $\alpha$ -cellulose, methyl cellulose, hydroxyethyl cellulose and cellulose acetate) as a precursor. First, the samples were carbonized under Ar atmosphere (at 400 or 500 °C), and then under CO<sub>2</sub> atmosphere for 2 h. Based on experimental results, the difference in functional groups in the cellulose samples not only affects the pore structure, but also improves the adsorption properties. As can be seen from the studies summarized above, activated carbons obtained from cellulose-rich precursors are better due to properties such as pore structure, pore size, surface area of activated carbon, and versatile surface functional groups.

One way to obtain cellulose-rich precursors is to delignify the biomass. There are various physico-chemical pretreatment methods for the delignification of lignocellulosic biomass such as acid pretreatment, NaOH pretreatment, ammonia pretreatment, organosolv pretreatment, ionic liquid pretreatment, deep eutectic solvent pretreatment (27). Among them, organosolv pretreatment has the advantage of lignin

recovery while enriching cellulose in the pulp. In the organosolv treatment, lignocellulosic biomass is brought into contact with a solvent in the presence of a catalyst (28). This process can be carried out under pressure, or it can be carried out at atmospheric pressure at relatively higher temperatures. The type of solvent and biomass, particle size of the biomass, biomass/solvent ratio, pretreatment conditions (temperature, pressure, mixing speed, time) affect the extent of delignification (29). The cellulose-rich fraction is separated from the solution by filtration. Lignin is precipitated by diluting the organosolv solution with water. Solvent recovery is often attempted from the solution if the solvent has low boiling point temperature (30). Sometimes hydrothermal pretreatment is applied prior to organosolv pulping to recover the hemicellulose sugars, to increase the accessibility of enzymes to cellulose and to boost delignification (31-35). Hydrothermal pretreatment, which is a simple and low-cost method, uses water as a solvent and pretreatment is carried out at temperatures between 160-230 °C (36). In this method, where most of the hemicellulose is degraded, swelling and partial hydrolysis of cellulose and partial decomposition and repolymerization of lignin are observed depending on the conditions of the hydrothermal pretreatment (37). After hydrothermal pretreatment, the liquid fraction containing a high percentage of sugar can be converted into platform chemicals using different conversion methods (38). The solid fraction, which is named as hydrochar, can be used as an adsorbent for environmental applications, a solid fuel for the production of energy or energy carriers (such as syngas), a precursor for the preparation of activated carbon or can be further processed to produce high value-added products (39, 40).

There are many studies in the literature that focuses on using cellulose, microcrystalline cellulose, or cellulose-rich biomass as precursors for activated carbon preparation. Studies which use cellulose-rich pulp from organosolv treatment as a precursor are very rare. Unlike the others, in this study, delignification of lignocellulosic biomass was done by a single-stage glycerol-organosolv treatment and a sequential hydrothermal pretreatment-organosolv treatment separately. Cellulose-rich pulps obtained by both methods and the tea stalk itself were used as precursors to produce microporous-mesoporous activated carbon. Activated carbons are compared based on their properties and potential end-uses were discussed.

## 2. EXPERIMENTAL SECTION

### 2.1. Materials

Tea stalk samples were supplied from a tea processing factory. All tea stalk samples were dried (105 °C for 24 h) before analyses and pretreatments to prevent samples from rotting and to provide better conditions for

storage. After grinding, samples were sieved by using RESTCH AS 200 vibrating sieve to have a uniform particle size (250  $\mu\text{m}$ ). Dry tea stalk samples with uniform particle size are named as TS.

Chemicals including glycerol, NaOH and  $\text{H}_2\text{SO}_4$  were of analytical grade and used without further purification.

## 2.2. Methods

### 2.2.1. Hydrothermal pretreatment

Hydrothermal pretreatment of TS was carried out in a 250 mL non-stirred, temperature-controlled, stainless-steel batch reactor. Hydrothermal pretreatment conditions were selected based on our previous study (pretreatment temperature: 220  $^{\circ}\text{C}$ , residence time: 90 min, tea stalk/water ratio: 1 g/4 mL) (41). After pretreatment, the reactor was cooled rapidly by using ice bath. Gases, which formed during the pretreatment were not collected. The mixture was filtered to separate hydrochar. The hydrochar of TS samples (named as HTS) were washed with deionized water and then dried in an oven at 105  $^{\circ}\text{C}$  for 24 h.

HTS samples were delignified by using alkaline glycerol organosolv treatment as explained below. The pulp obtained after sequential hydrothermal pretreatment and organosolv treatment was named as HTP.

### 2.2.2. Alkaline-glycerol organosolv treatment

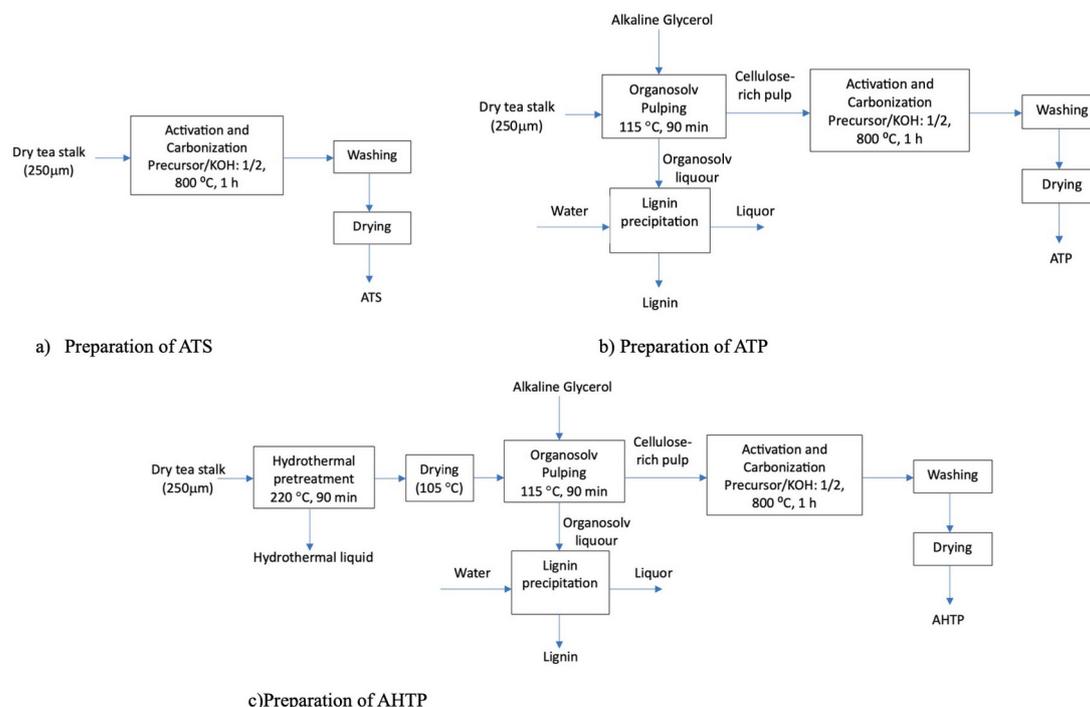
Alkaline-glycerol organosolv method, which we previously used for delignification (42), was modified in this study to increase the lignin yield. 20 g tea stalk, 50

mL of 0.4 M NaOH solution and 150 mL glycerol were mixed in a three-necked glass reactor equipped with a reflux condenser and a thermocouple. First, the mixture was mixed for 30 min at 90  $^{\circ}\text{C}$ , then the temperature was raised to 115  $^{\circ}\text{C}$ . The mixture was continuously mixed for 90 min at this temperature. At the end of the treatment, pulp was separated from the liquor, and washed with hot water (water at 55  $^{\circ}\text{C}$ ) and then dried in an oven at 100  $^{\circ}\text{C}$  for 24 h. The pulp obtained after organosolv pretreatment was named as TP.

To precipitate the lignin, filtrate was diluted with deionized water to 1L (at 55  $^{\circ}\text{C}$ ) and the pH was decreased to 2-2.5 by using 0.1 M  $\text{H}_2\text{SO}_4$ . After precipitation, lignin was separated, washed with water and then dried. Characteristics of lignins are not given as they are beyond the scope of this article.

### 2.2.3. Preparation of activated carbon

Dry TS, TP and HTP samples were mixed with KOH at a ratio of 1:2 (wt:wt). After gently mixing KOH with the sample, the mixture in a ceramic boat was placed in a horizontal tubular furnace. The sample was heated at a heating rate of 10  $^{\circ}\text{C}/\text{min}$  up to 800  $^{\circ}\text{C}$  and kept at this temperature for 1h. During heating and cooling, tubular furnace was continuously flushed with nitrogen gas. After activation, samples were washed with 0.1 M HCl and deionized water. Then, the samples were dried at 105  $^{\circ}\text{C}$  for 24 h. Activated carbons obtained from TS, TP and HTP were named as ATS, ATP and AHTP respectively. Block diagrams regarding the preparation of ATS, ATP and AHTP are given in Figure 1.



**Figure 1:** Preparation procedure and naming of the activated carbons.

## 2.3. Analyses

Proximate analyses of TS, HTS, TP and HTP were done using thermogravimetry method as described in Garcia,

Pizarro (43). The elemental analysis of TS, HTS, TP, HTP, ATS, ATP and AHTP were carried by an elemental analyzer (LECO, CHNS-932). Acid-insoluble lignin content of TS, TP and HTP was determined by using gravimetric method as described in NREL/TP-510-42618 (44). Holocellulose content of TS was determined as explained in Salim et al. (45).

Fourier Transform Infrared (FTIR) spectra of TS, HTS, TP, HTP, ATS, ATP and AHTP were obtained using a spectrometer (Perkin Elmer Spectrum 100) coupled with a universal attenuated total reflectance (ATR) sampling device with a diamond crystal. The spectra were recorded with a resolution of 4 1/cm, in the range from 400 to 4000 1/cm.

For thermal stability, thermogravimetric analyzer (Seiko, TG/DTA 6300) was used. Samples were heated from room temperature to 900 °C with a heating rate of 40 °C/min and maintained for 7 min.

Surface morphologies of HTS, ATS, ATP and AHTP were investigated by FEI Inc., Inspect S50 SEM. Samples were investigated under 20 kV in the high vacuum mode using secondary electrons with different magnifications.

The surface area and pore structure characteristics of ATS, ATP and AHTP were determined by using Quantachrome-Autosorb iQ BET analyzer at 77K in the relative pressure range of 0.001 and 0.99. Before analysis, samples were degassed at 180 °C for 24 h.

The XRD patterns of ATS, ATP and AHTP were collected using Rigaku Smartlab X-Ray diffractometer, operating at 40kV and 30 mA, with a scan range of 10 to 80 °, a step width of 0.02° and a scan speed of 1.0039 °/min.

### 3. RESULTS AND DISCUSSION

#### 3.1. Characterization

The proximate analysis (volatile matter, fixed carbon and ash content) and ultimate analysis (C, H, N and S content) of TS, HTS, TP, HTP, ATS, ATP and AHTP are presented in Table 1. Tea stalk is a lignin-rich lignocellulosic biomass, which contains 33.9±1.1 % lignin and 49.2±1.3 holocellulose in its structure.

During hydrothermal pretreatment, biomass undergoes several reactions including hydrolysis, dehydration, decarboxylation, condensation, and polymerization (40). The extent of the reactions, which determines the hydrochar yield (mass ratio of hydrochar to biomass on dry weight basis), depend on the hydrothermal conditions and the type of biomass (especially lignin, cellulose and hemicellulose content). After hydrothermal pretreatment of TS, the hydrochar yield was calculated as 70.7±0.3 %. Due to the high lignin content of TS and moderate pretreatment temperature (220 °C), this hydrochar yield was expected as explained briefly in Gulec et al. (46).

After hydrothermal pretreatment, the volatile matter content of the tea stalk increased, the fixed carbon content decreased, and consequently the carbon content increased by 3%. Hydrothermal pretreatment also reduced the ash content of the TS by 5.3%.

Alkaline-glycerol organosolv pretreatment is an effective delignification method (28). When alkaline-glycerol organosolv treatment was applied directly to TS, 7.1±0.2 % lignin (acid-insoluble lignin) was detected in TP, showing that some of the lignin retained in the pulp. In the case of sequential hydrothermal pretreatment and organosolv treatment, the lignin content in HTP was 11.6±0.7 %. Based on acid-soluble lignin content, hydrothermal pretreatment prior to glycerol organosolv treatment reduced delignification.

The application of hydrothermal pretreatment before the organosolv treatment affected the pulp content. The carbon content of TP and HTP was close to each other (37.8% and 36.9%, respectively) and a small part of this carbon is fixed carbon. Cellulose-rich TP and HTP were found to have a lower carbon content than the average carbon content of a typical cellulose (44.06 %), and a higher fixed carbon content than a typical cellulose (4.35 %) (47). Since delignification was not 100% in both treatments, there was still lignin in both TP and HTP. The high fixed carbon content of TP and HTP can be explained by the high fixed carbon content of the residual lignin (23.09 %) (47).

**Table 1:** Proximate and ultimate analysis of TS, HTS, TP, HTP, ATS, ATP and AHTP.

	TS	HTS	TP	HTP	ATS	ATP	AHTP
<b>Proximate analysis (wt.%, db.)</b>							
VM	71.1	72.6	91.4	92.3	n.d.	n.d.	n.d.
FC	25.1	23.8	6.9	6	n.d.	n.d.	n.d.
Ash	3.8	3.6	1.7	1.7	n.d.	n.d.	n.d.
<b>Ultimate analysis (wt.%, db.)</b>							
C	46.6	48	37.8	36.9	61.3	56.3	52.3
H	5.5	5.4	8.3	8	0.5	0.2	0.2
N	2.6	2.3	0.5	0.6	1.1	0.5	0.4
S	0.1	0	0	0	0	0	0

VM: Volatile matter, FC: Fixed carbon, A: Ash, db: dried basis

\* Proximate and ultimate analyses were done twice. Mean values were given in Table 1.

After activation-carbonization, the carbon content of TS, TP and HTP increased by 31.5%, 49% and 41.7%, respectively. Based on elemental analysis results, the oxygen content of ATP and AHTP was high. This was mainly due to high oxygen content of TP (51.7%) and HTP (52.8%). Not hydrothermal pretreatment but delignification definitely increased the oxygen content of the pulps. The oxygen-rich functional groups of TP, HTP, ATP and AHTP were also supported by the FTIR spectra.

### 3.2. Thermal Stability of TS, HTS, TP and HTP

Thermal stability is an important property of all bio-based materials. Figure 2 shows the TG and DTG curves of TS, HTS, TP and HTP. The thermogravimetric curve of TS showed a thermal degradation curve of a typical biomass. Moisture removal was observed in the first stage at temperatures between 25 °C and 110 °C. The thermal degradation of structural components (hemicellulose, cellulose and lignin) was seen at temperatures between 120 °C and 600 °C.

Like TS, the thermal degradation of HTS occurred in two stages at a wide temperature range. Hydrochar has a more stable structure compared to biomass since hemicellulose is partially degraded and light volatiles leave the structure during hydrothermal treatment. HTS was also more thermally stable than TS. Better thermal stability of the structure was also supported by the shifting of  $T_{max}$  (the temperature at which the mass loss

rate is maximum) from 337 °C to 344 °C after hydrothermal treatment.

While the thermal degradation of pure cellulose takes place in two stages (the first step is moisture removal), the thermal degradation of TP and HTP differs slightly from the typical thermogram of cellulose. This is because although it is rich in cellulose, there is some lignin in the pulp and chemical treatment of the cellulose disrupts the structure (48). Studies investigating the effects of chemical treatment on cellulose structure have shown that the thermal degradation of cellulose occurs in three stages, and the thermal degradation after moisture removal is between 110 °C – 285 °C and 288 °C – 600 °C (49, 50). In the present study, thermal degradation of both TP and HTP occurred in three steps. The mass loss in the first stage in both samples was due to the removal of moisture. The degradation of TP in the second stage was between 180 °C and 235 °C, with a  $T_{max}$  of 220 °C. The mass loss of TP in the third stage was between 280 °C and 344 °C, and the highest mass loss rate in this stage was seen at 322 °C.

The thermal degradation of HTP started with the removal of moisture. The second degradation was between 172 °C and 236 °C, with a  $T_{max}$  of 221 °C. The third stage of thermal degradation was observed at temperatures between 290 °C and 344 °C. At this stage, the temperature with the highest mass loss rate was determined as 327 °C. Application of hydrothermal pretreatment before the organosolv treatment slightly increased the thermal stability of the pulp.

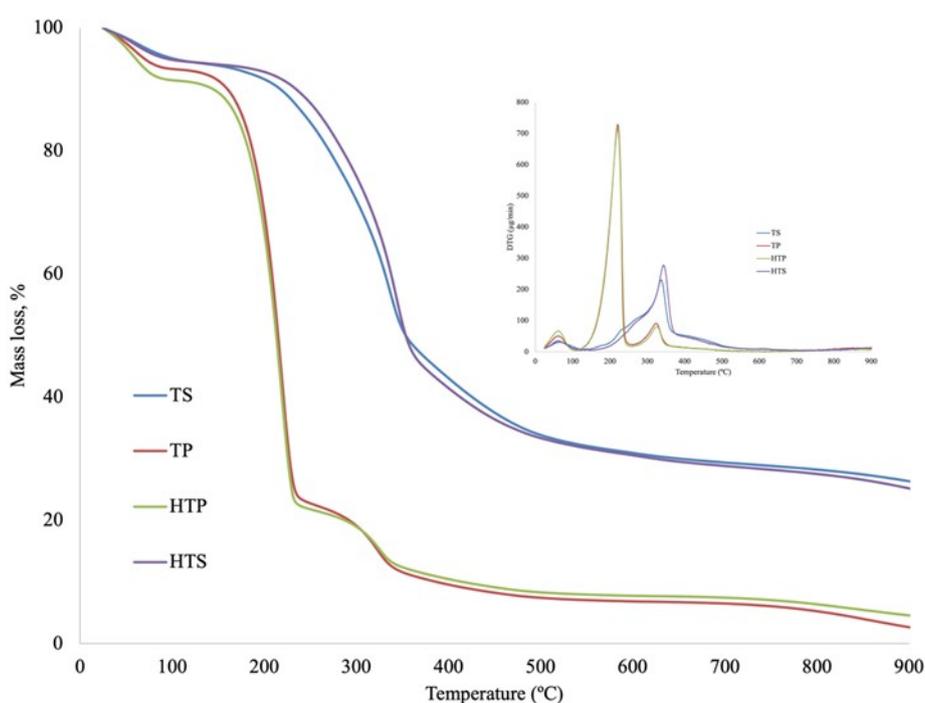


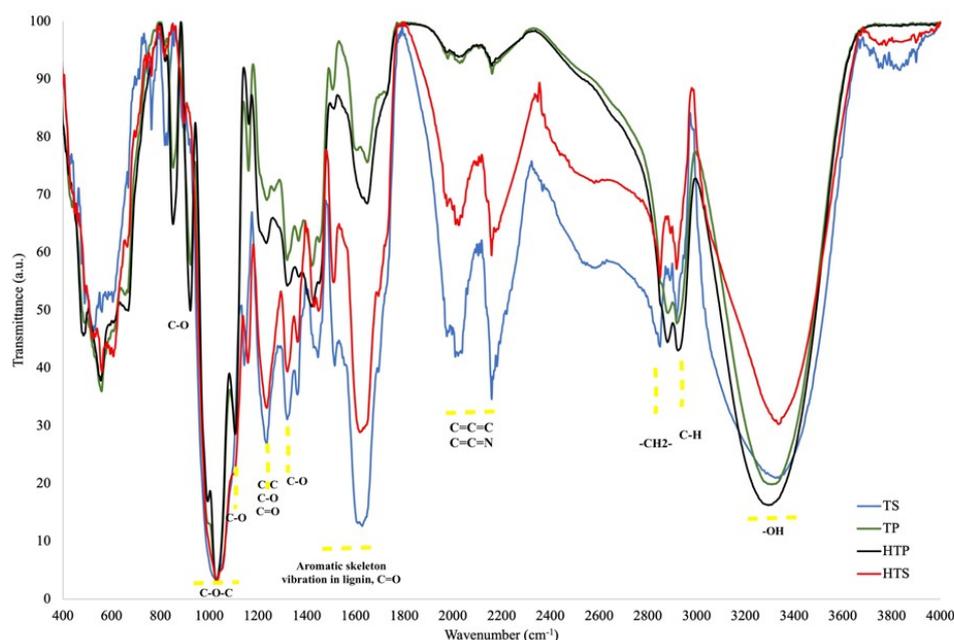
Figure 2: TG curves of TS, HTS, TP and HTP.

### 3.3. FTIR Analysis

The FTIR spectra of TS, HTS, TP and HTP are shown in Figure 3. Tea stalk has a broad peak at 3000-3800 1/cm (centered at 3338 1/cm) due to -OH stretching vibration in the hydroxyl functional groups (in compounds such as phenolics, aliphatic alcohols, carboxylic acids etc.) (51). The peaks at 2917 1/cm and 2849 1/cm correspond to stretching vibration of aliphatic C-H bond in  $-CH_2-$  structures (52). The peaks appearing between 2009-2160 1/cm originate from the stretching vibration of allene ( $C=C=C$ ) and ketamine ( $C=C=N$ ) groups in tea stalk (51, 53, 54). Stretching vibration of  $C=C$  bond in aromatic rings and C-H stretching appear at 1515 1/cm and 1362 1/cm. HTS has a similar FTIR spectra with that of TS. Only difference between them was the intensity of the bands of HTS at 1200-3600 1/cm region, which confirms that a degradation was observed but functional group was not changed. Compared to TS, the stretching vibration of -OH (at 3000-3600 1/cm), -CH (at 2800-3000 1/cm) and  $C=C=C$  (at 2000-2200 1/cm) in HTS structure were much lower. After hydrothermal pretreatment, the characteristic bands of lignin (1660-1638 1/cm) and (1400-1500 1/cm) were retained, with a slight decrease in intensities. Yu et al. (55) made a similar observation and concluded that the structure of

lignin was slightly changed with temperature due to the cleavage of C-C, C=C and C-H bands. The characteristic stretching vibrations of cellulose at 1000-1400 1/cm region were slightly less due to partial degradation of cellulose under hydrothermal conditions.

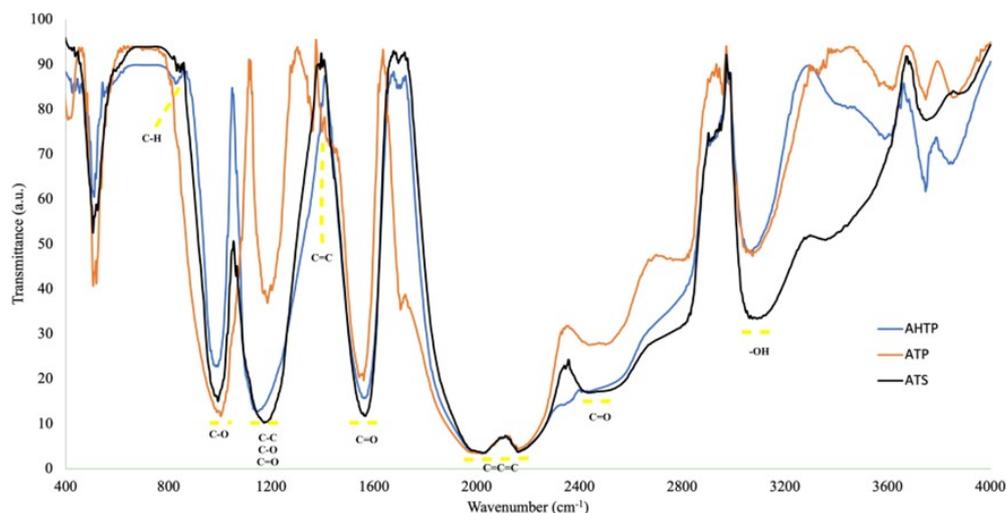
The effect of delignification was observable from the FTIR spectra of TP and HTP. The wide peak appearing between 3000-3600 1/cm for all samples reflect the stretching vibration of -OH group. The bands at 2880 1/cm and 2920 1/cm were assigned to -CH stretching vibration of  $-CH_2$  and  $-CH_3$  groups. The peaks at 2160 1/cm, 2040 1/cm and 2000 1/cm were attributed to  $C=C=C$  and  $C=C=N$  stretchings. While FTIR spectra of TS reflects the conjugated carbonyl stretching (at 1660-1638 1/cm) and aromatic skeleton vibration in lignin (at 1600 1/cm, 1500 1/cm and 1440 1/cm) (56, 57); the intensity of the same peaks in the FTIR spectra of both TP and HTP were comparatively very low due to delignification. In all spectra, in-plane C-H deformation (at 1360 1/cm), in-plane O-H bending in cellulose structure (at 1320 1/cm) and C-C, C-O, C=O stretch (at 1240 1/cm) were observed.



**Figure 3:** FTIR spectra of TS, HTS, TP and HTP.

Figure 4 shows the FTIR spectra of ATP, AHTP and ATS. The spectra of the activated carbons were similar, only the intensities of the vibrations altered. The stretching vibration of -OH in phenol and carboxyl groups centered at 3073 1/cm (58) was more intense in the spectra of ATS than that of ATP and AHTP. Similarly, C=O stretching vibration (in ketene groups) at 2420-2430 1/cm (59) was more intense in the spectra of ATS than that of ATP and AHTP. The spectra of all activated carbons showed the stretching vibration of  $C=C=C$  (in allene group) at 2160 1/cm (60, 61). C=O stretching

vibration of carbonyl and carboxylate groups (such as ketones, aldehydes) was observed in the spectra of all activated carbons at 1600-1700 1/cm with same intensity (62).  $C=C$  stretching vibration of aromatic rings at 1560 1/cm (51) was also detected in all spectra. C-O stretching vibration (in ethers, alcohols, acids, esters) at 1017 1/cm (63) was much more intense in the spectra of ATP. AHTP and ATS showed stronger stretching at 1200 (C-C, C-O, C=O stretching vibration). Bending vibration of C-H in the aromatic rings at 820-840 1/cm (64) was observed in the spectra of AHTP and ATS.



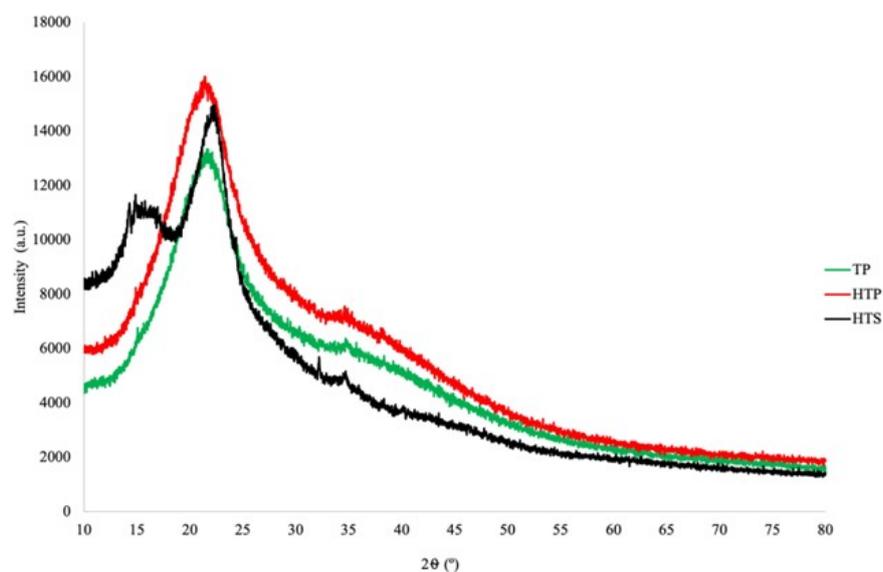
**Figure 4:** FTIR spectra of ATS, ATP and AHTP.

### 3.4. X-ray Diffraction Analysis

Depending on the type of biomass, cellulose is present in the structure as crystalline and amorphous form (65). Any pretreatment using chemicals such as acids, bases and solvents might affect the amorphous structure (66-70). In order to observe what extent the pretreatments changed the cellulose in the biomass structure, x-ray diffraction analysis was also performed on hydrochar and pulps.

XRD patterns of TP, HTP and HTS are presented in Figure 5. The XRD patterns of HTS exhibit diffraction peaks at  $15^\circ$  (101),  $22^\circ$  (002) and  $34.5^\circ$  (040), which indicates that both amorphous and crystalline cellulose structure was preserved during hydrothermal pretreatment (64). A similar result was observed by Fan et al. (71), who studied the hydrothermal carbonization

of Chinese fan palm leaves at different temperatures. In the study of Fan et al. (72), a broad peak between  $15^\circ$  and  $16^\circ$ , which indicated amorphous structure, was observed in the XRD of hydrochars obtained at 180 and  $220^\circ\text{C}$ , while this structure was not seen above  $260^\circ\text{C}$ . In the x-ray diffractions of TP and HTP obtained by organosolv pretreatment and sequential hydrothermal-organosolv pretreatments, only broad peaks at  $22^\circ$  (002 plane) and  $34.5^\circ$  were observed. Alkaline-glycerol organosolv pretreatment completely removed the amorphous structure. Sun et al. (72), who applied glycerol organosolv treatment to wheat straw, showed that the amorphous structure (amorphous cellulose, lignin and hemicellulose) decreased (hence the crystal index increased) and the average crystal size of cellulose decreased.

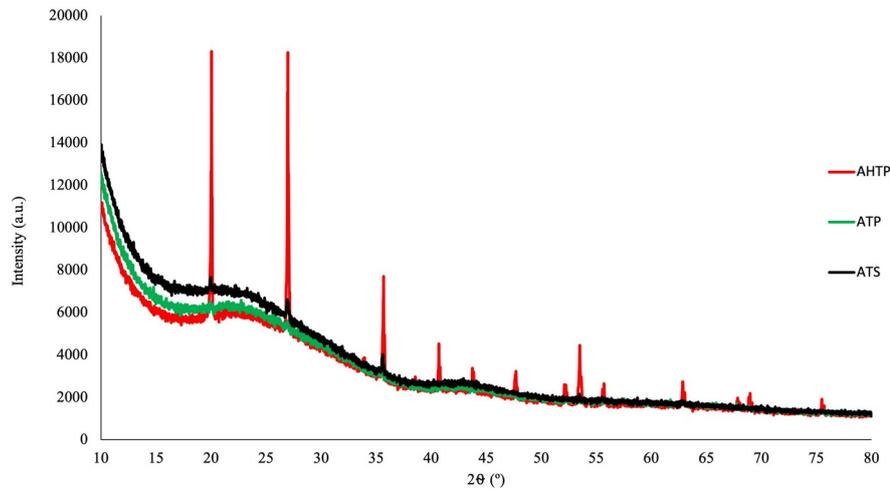


**Figure 5:** X-ray diffraction patterns of TP, HTP and HTS.

After carbonization and activation, a broad hump (indicating the amorphous graphitic carbon) between  $17^\circ$  and  $35^\circ$  was observed in the x-ray diffraction pattern of ATP, ATS and AHTP (73). A weaker and

broader hump at  $41-47^\circ$  was also observable in all patterns, which is also a characteristic diffraction of amorphous graphite structure (74). Unlike ATP and ATS, AHTP shows sharp diffraction peaks of graphitic carbon

at  $2\theta=20^\circ, 26^\circ, 36^\circ, 41^\circ, 44^\circ, 47.5^\circ, 52^\circ, 53.5^\circ, 55.5^\circ, 63^\circ$  (55, 75). Obviously, AHTP has higher crystallinity compared to ATP and ATS.

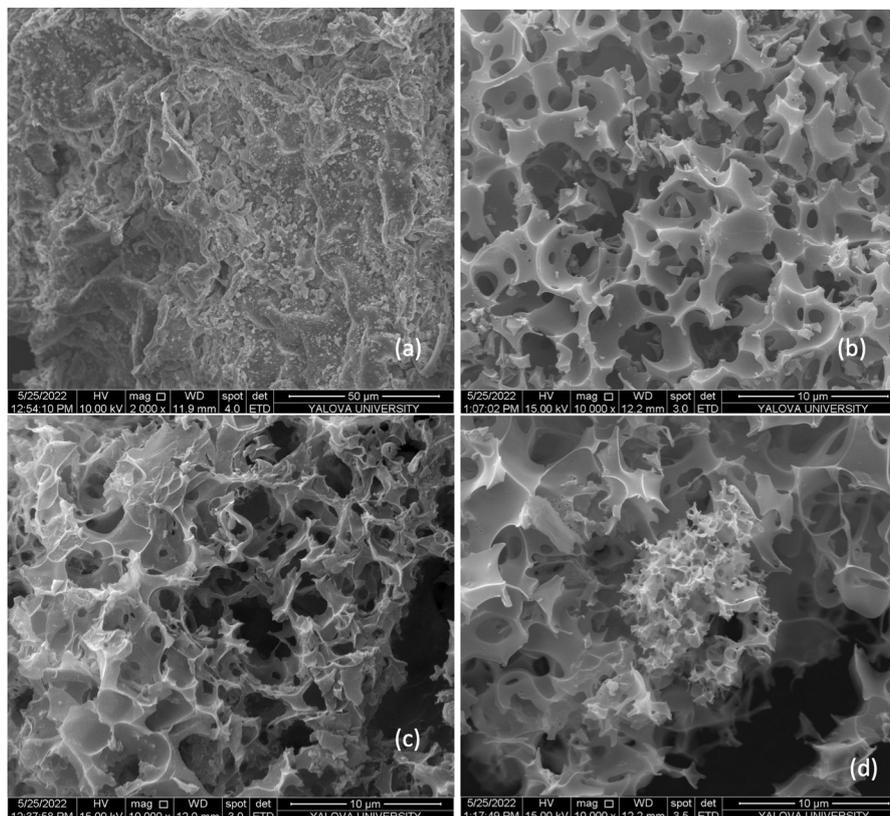


**Figure 6:** X-ray diffraction pattern of AHTP, ATP and ATS.

### 3.5. SEM Analysis

SEM provides micrographs that show surface morphology. The surface morphologies of HTS, ATS, ATP and AHTP can be seen in Figure 7. HTS shows an irregular and rough surface. The formation of microspheres or pores was not observed on the surface of HTS, showing that hydrothermal treatment temperature ( $220^\circ\text{C}$ ) was not able to destruct lignocellulosic structure (76). The SEM image of ATS shows a dense porous structure with many three-dimensional channels. Activation of tea stalk with KOH resulted in well-developed micropores on the surface of

the ATS. Although the SEM image of ATP was densely microporous like that of ATS, the presence of mesopores was noteworthy. Removal of lignin from the lignocellulosic structure leads pore formation. Therefore, mesopores in ATP were due to severe destruction of lignocellulosic structure by organosolv delignification, which was done prior to activation. AHTP images also show the porous structure with a wider range of pores compared to that of ATP. Both hydrothermal pretreatment and organosolv delignification corroded the lignocellulosic structure, which resulted in mesopore formation.



**Figure 7:** SEM images of (a) HTS (magnification of 2000x), (b) ATS, (c) ATP and (d) AHTP (magnification of 10000x).

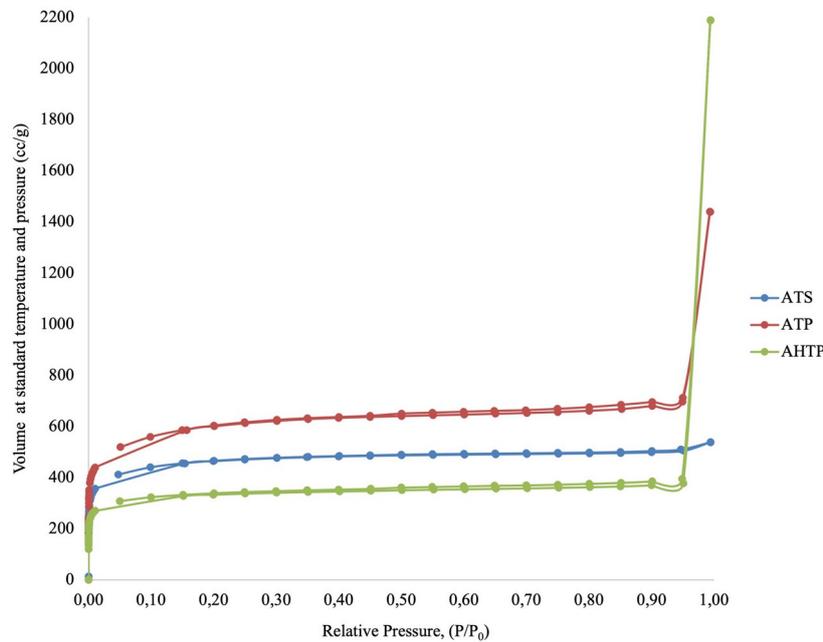
### 3.6. BET Analysis

To have high adsorption capacity, activated carbons should possess morphological properties including amorphous structure, high surface area and high porosity (1). Obtaining  $N_2$  adsorption-desorption isotherms is the most common method used to determine the surface area and pore characteristics of activated carbons. Pores can be classified as micropores (pore diameter <2 nm), mesopores (pore diameter between 2-50 nm), and macropores (pore diameter > 50 nm) (77).

Isothermal plots of ATP, AHTP and ATS are shown in Figure 8.  $N_2$  adsorption-desorption isotherm of ATS is Type I isotherm (based on IUPAC classification), showing microporous characteristics.  $N_2$  isotherm of ATS shows a steep  $N_2$  uptake at very low relative pressures ( $P/P_0 < 0.1$ )

due to  $N_2$ -ATS interactions in narrow micropores by micropore filling (78).

Both ATP and AHTP show Type IV isotherm, with a steep uptake of  $N_2$  at very low relative pressures and a sharp increase of  $N_2$  adsorption at relative pressures higher than 0.9. ATP and AHTP are mesoporous activated carbons. Adsorption of  $N_2$  in mesopores is due to physical interaction between  $N_2$  molecules and activated carbon, and the interaction between  $N_2$  molecules in the condensed state. Since AHTP has larger pores,  $N_2$  adsorption due to condensation is higher. For both AHTP and ATP, hysteresis due to capillary condensation is not observed in the isotherm, showing that mesopores are either smaller in width or in conical/cylindrical form (78).

**Figure 8:** Nitrogen adsorption/desorption isotherms of ATS, ATP and AHTP.

The surface area and pore structure characteristics of ATS, ATP and AHTP are listed in Table 2. ATP has the highest surface area (2056.72  $m^2/g$ ), where 86% of it comes from micropores. This is mainly due to the porosity enhancement by delignification (35, 79) and better accessibility of KOH to the active sites. Similar results were observed in studies that produced activated carbon from cellulose-rich precursors. Tsubota

et al. (21) first delignified bamboo powder with acetic acid-hydrogen peroxide solvent mixture and then carbonized the pulp in the presence of  $CO_2$  at 800 °C. Regardless of the lignin content of the pulp, the surface areas of activated carbons varied between 1062.88  $m^2/g$ -1413  $m^2/g$ , and the average pore sizes varied between 1.7-2 nm.

**Table 2:** Comparison of surface area and pore characteristics.

Sample	$S_{BET}$ ( $m^2/g$ )	$S_{micropore}$ ( $m^2/g$ )	$S_{micropore}/S_{BET}$	Average pore radius (nm)	$V_{micro}$ ( $cm^3/g$ )	$V_{total}$ ( $cm^3/g$ )
ATS	1643	1479.14	0.90	1.01	0.65	0.833
ATP	2056.72	1768.66	0.86	2.16	0.81	2.227
AHTP	1179.71	1055.53	0.89	5.74	0.46	3.386

$S_{BET}$ : Surface area based on multi-point BET analysis

$S_{\text{micropore}}$ : micropore area based on V-t method

$V_{\text{micro}}$ : micropore volume

AHTP, with the largest average pore radius (5.74 nm), has the least micropore volume (0.464 cm<sup>3</sup>/g) and surface area (1179.71 m<sup>2</sup>/g). Sequential hydrothermal pretreatment and organosolv treatment destruct the lignocellulosic structure more resulting in a more porous precursor, which ended with mesoporous structure. ATS, having the lowest average pore radius, is microporous. Activated carbons of different lignocellulosic biomass (which are activated by KOH and carbonized at 800 °C) have similar pore diameter and surface area with ATS (80-84).

### 3.7. Possible Applications of the Microporous and Mesoporous Activated Carbons Prepared from Tea Stalk and Tea Stalk Pulp

This study showed that although the same activation and carbonization conditions are applied, pretreatments namely organosolv delignification and two-step hydrothermal-organosolv treatment affect the pore diameters, surface functional groups and specific surface areas of activated carbons. Nonetheless, those characteristics of activated carbons are important in determining the end use or application. Since the specific surface areas and pore structures of the prepared ATS, ATP and AHTP are different, the most appropriate end-use purpose of those activated carbons should differ. Possible uses are discussed below, considering the specific surface area and pore structure.

One of the processes in which activated carbon is used the most is adsorption. While micropores provide the main adsorption sites, mesopores provide diffusion channels for the adsorbate (85). In liquid and gaseous adsorption processes, the pore size of the adsorbent and the molecular size of the adsorbate must be compatible (85). Due to the reason that gaseous adsorbate molecules are generally found in the range of 0.5–1.5 nm, the microporous activated carbons (average pore size <2 nm) are suitable for the adsorption of gases (86). Microporous activated carbons are mainly used for the adsorption of volatile organic compounds (VOCs), CH<sub>4</sub>, N<sub>2</sub>, and CO<sub>2</sub> (87). Since the presence of oxygen-containing functional groups lowers the adsorption capacity (88, 89), oxygen-rich microporous ATS might not be suitable to be used for the adsorption of VOCs. In the case of CH<sub>4</sub> and N<sub>2</sub> adsorption, it has been shown in studies that the pore size should be quite low (between 0.7 nm and 1.3 nm) for high adsorption capacity (86, 90, 91). Many experimental studies related to CO<sub>2</sub> adsorption show that both the preparation method and structural properties of the activated carbons are similar to ATS. The high specific surface area, micropore structure and presence of highly oxygenated functional groups of ATS make ATS a suitable candidate for CO<sub>2</sub> adsorption. Table 3 shows the structural properties of activated carbons and their CO<sub>2</sub> adsorption capacities that were obtained under similar conditions to our study.

**Table 3:** Structural properties of activated carbons and their CO<sub>2</sub> adsorption capacities reported in similar studies.

Precursor	Carbonization and activation conditions	Surface area (m <sup>2</sup> /g)	Pore size (nm)	Micropore volume (cm <sup>3</sup> /g)	CO <sub>2</sub> adsorption	Ref.
Pine nut shell	KOH/precursor: 2(wt ratio) T: 800 °C	1372	2.1532	0.6842	5.8 mmol/g (0 °C) 3.5 mmol/g (25 °C)	(92)
Walnut shell	KOH/precursor: 1(wt ratio) T: 800 °C	1868	< 4	0.55	9.56 mmol/g (0 °C) 5.17 mmol/g (25 °C) 4.34 mmol/g (40 °C)	(93)
Cucumber peels	KOH/precursor: 1(wt ratio) T: 800 °C	1769	2.334	0.692	6.095 mmol/g (0 °C) 4.37 mmol/g (10 °C) 3.52 mmol/g (25 °C)	(94)
Starch	KOH/precursor: 4 (wt ratio) T: 700 °C	2190	1.2	0.92	5.6 mmol/g (0 °C) 3.5 mmol/g (25 °C) 5.6 mmol/g (50 °C)	(95)
Cellulose	KOH/precursor: 4 (wt ratio) T: 700 °C	2370	1.2	0.96	5.8 mmol/g (0 °C) 3.5 mmol/g (25 °C) 1.8 mmol/g (50 °C)	
Sawdust	KOH/precursor: 4 (wt ratio) T: 700 °C	2250	1.2	0.91	5.5 mmol/g (0 °C) 2.9 mmol/g (25 °C) 1.8 mmol/g (50 °C)	
Sawdust	KOH/precursor: 2 (wt ratio) T: 800 °C	1940	0.9	0.82	5.8 mmol/g (0 °C) 3.9 mmol/g (25 °C) 3.1 mmol/g (50 °C)	(96)
Olive mill waste hydrochar	KOH/precursor: 2 (wt ratio) T: 700 °C	888	< 1	0.294	1.350 mmol/g (0 °C, 1 bar) 2.984 mmol/g (0 °C, 101.3 kPa)	

Camphor leaves	KOH/precursor: 2 (wt ratio) T: 800 °C	1736	< 1	0.357	4.8 mmol/g (0 °C) 2.42 mmol/g (25 °C) 1.45 mmol/g (50 °C)	(97)
Pristine wheat flour	KOH/precursor: 2 (wt ratio) T: 700 °C	1057	< 2	0.474	4.41 mmol/g (0 °C) 2.77 mmol/g (25 °C) 1.60 mmol/g (50 °C)	(98)
Peanut shell	KOH/precursor: 1 (wt ratio) T: 700 °C	956	<11	Not available	1.54 mmol/g (25 °C, 0.15 bar)	(99)
Sunflower seed shell	KOH/precursor: 1.25 (wt ratio) T: 700 °C	1790	< 1	Not available	1.46 mmol/g (25 °C, 0.15 bar)	
Pine nut shell	KOH/precursor: 2 (wt ratio) T: 700 °C	Not available	<1.1	Not available	7.7 mmol/g (0 °C, 1 bar) 5 mmol/g (25 °C, 1 bar) 3.3 mmol/g (0 °C, 0.15 bar) 2 mmol/g (25 °C, 0.15 bar)	(100)
Paulownia sawdust	KOH/precursor: 2 (wt ratio) T: 700 °C	831	<1	0.297	6.83 mmol/g (0 °C)	(101)
Paulownia sawdust	KOH/precursor: 4 (wt ratio) T: 800 °C	1555	<1	0.598	7.14 mmol/g (0 °C)	
Spent coffee grounds	KOH/precursor: 3 (wt ratio) T: 700 °C	1082	3	0.44	3.2 mmol/g (0 °C, 101 kPa) 2.7 mmol/g (25°C, 101 kPa)	(102)

Mesoporous activated carbons (2 nm < average pore size <50 nm) are more preferred in the adsorption of large molecules including dyes, pharmaceuticals,

cyanobacterial toxins, pollutants and enzymes (103-105).

**Table 4:** Structural properties of mesoporous activated carbons and their reported adsorbates and adsorption capacities reported in similar studies.

Precursor	Carbonization and activation conditions	Surface area (m <sup>2</sup> /g)	Pore size (nm)	Micropore volume (cm <sup>3</sup> /g)	Adsorbate and adsorption capacity	Ref.
Dragon fruit peels	KOH/precursor: 2 (wt ratio) T: 700 °C	756.3	11.3	0.376	Adsorbate: methylene blue dye Maximum adsorption capacity: 195.2 mg/g	(106)
Yellow mombin fruit stones	KOH/precursor: 1 (wt ratio) T: 500 °C	167	4.82	0.027	Adsorbate: Dianix ® royal blue dye Maximum adsorption capacity: 82.28 mg/g	(107)
Rice straw	Precursor was soaked in 85 wt % KOH solution T: 400 °C for 1 h and then 700 °C for 1 h.	165.9	5.941	0.032	Adsorbate: Methylene blue dye Maximum adsorption capacity: 392.4 mg/g Adsorbate: Congo red dye Maximum adsorption capacity: 178.4 mg/g	(108)
Rice straw	Precursor was first pre-carbonized at 400 °C for 1 h, then soaked in 85 wt% KOH solution, then carbonized at 700 °C for 1 h.	1973	2.292	0.769	Adsorbate: Methylene blue dye Maximum adsorption capacity: 527.6 mg/g Adsorbate: Congo red dye Maximum adsorption capacity: 531.4 mg/g	(109)
Sugarcane bagasse	KOH/precursor: 1 (wt ratio) T: 700 °C	1204	1.79	0.57	Adsorbate: Phenol Maximum adsorption capacity: 88 mg/g	(109)
Sawdust	KOH/precursor: 1 (wt ratio) T: 700 °C	1544	1.82	0.69	Adsorbate: Phenol Maximum adsorption capacity: 96.8 mg/g	(109)
Potato peel	KOH/precursor: 4 (wt ratio) T: 600 °C	2394	<10 Average between: 2-4 nm	-	Adsorbate: Methylene Blue Maximum adsorption capacity: 2521 mg/g	(110)
Crab shell	KOH/precursor: 3 (wt ratio) T: 800 °C	1095.14	2.18	-	Adsorbate: Tetracycline Maximum adsorption capacity: 380.92 mg/g	(111)

Mesoporous activated carbons are also preferred as electrode materials in supercapacitors due to their high specific surface area, high pore volume, good thermal stability, and enhanced specific capacitance (112, 113). Apart from mesoporous structure, presence of oxygen-containing functional groups on the surface of the activated carbon also enhances electrochemical active sites (111). Table 5 shows the studies in which activated carbon was prepared under conditions similar to those

we applied in the present study and used in supercapacitors. According to the studies in Table 5, having pores with a diameter of 2-5 nm enhances the transport of electrolytes. The large specific surface area, the presence of macropores alongside micropores, and the abundance of oxygenated functional groups make ATP and AHTP a suitable candidate for use in supercapacitors.

**Table 5:** Structural properties of activated carbons and their maximum specific capacitance reported in similar studies.

Precursor	Carbonization and activation conditions	Surface area (m <sup>2</sup> /g)	Pore size (nm)	Pore volume (cm <sup>3</sup> /g)	Maximum specific capacitance (F/g)	Ref.
Corncob	Carbonization at 400 °C for 4 h, then activation with KOH (KOH/precursor: 3 (wt ratio) ) at 600 °C	800	Micropores in the range of 1.4-1.8 nm, mesopores in the range of 2.2-5 nm	0.337	390	(114)
Wastewater from vitamin C production, template: melamine foam	KOH/precursor: 5 (wt ratio) T: 700 °C	3837	2.32	2.22	217	(113)
Olive tree branches	Carbonization: 500 °C for 2 h Activation: KOH/precursor: 4 (wt ratio) 800 °C for 82 min	2980	0.8nm<pore size<5 nm	1.59	410	(115)
Biogas slurry	First drying, then carbonization at 650 °C for 1 h. Activation: KOH/precursor: 3 (wt ratio) 700 °C for 30 min	326.7	4.7	0.24	163	(116)
	First drying, then carbonization at 650 °C for 1 h. Activation: KOH/precursor: 3 (wt ratio) 700 °C for 60 min	514.7	4.7	0.38	182	
Coconut silk	Carbonization at 400 °C for 1 h. Activation: KOH/precursor: 4 (wt ratio) 900 °C for 1 h	2318	0.5- 5 (average 1.42)	0.87	631	(111)
Corn silk	Carbonization at 300 °C for 2 h. Activation: KOH/precursor: 3 (wt ratio) 850 °C for 4 h	2441	3.097 (average) (large pores (>30nm) were also seen in the structure)	1.890	174	(117)

#### 4. CONCLUSION

In the present study, three different activated carbons were prepared: activated carbon from tea stalk itself, activated carbon from tea stalk pulp obtained by using glycerol organosolv pretreatment, and activated carbon from tea stalk hydrochar pulp obtained by using sequential hydrothermal pretreatment-organosolv delignification. The tea stalk itself and the cellulose-rich precursors were carbonized at 800 °C for 1 h in the presence of KOH.

The study showed that partial removal of lignin yielded activated carbons with more specific surface area and mesopores. Additionally, because delignification modified the functional groups of the biomass, the surface of activated carbon contained more oxygen-rich functional groups. While decreasing the lignin removal, hydrothermal pretreatment prior to delignification

ended up with mesoporous activated carbon with a larger pore diameter. Activated carbons (ATS, ATP and AHTP) obtained from tea stalk can be used in many applications from environmental applications to energy storage due to their different surface areas, micropore volumes and pore structures.

#### 5. CONFLICT OF INTEREST

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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