Activated Carbon Prepared from Corn Biomass by Chemical Activation with Potassium Hydroxide

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With the depletion of fossil fuel feedstocks, the lignocellulosic biomass, including the agro-wastes, can serve as the best alternative source to produce activated carbons (ACs). Corn biomass (corn leaves, stalks, cobs without kernels, silk, and kernels) were used to produce ACs in a two-step process. Crushed plant material was carbonized at 600 °C and then the obtained carbon was activated using potassium hydroxide at 750 °C. The content and type of surface oxygen functional groups were determined by the Boehm method and infrared spectroscopy. The porous structure of the obtained AC was determined by the nitrogen adsorption/desorption method at -196 °C, and the thermal resistance by the thermogravimetric method. The iodine number was also determined. The ACs derived from corn biomass were characterized with surfaces rich in chemical groups and revealed a highly developed porous structure. The specific BET surface area ranged from 1600 m^2/g to 1965 m^2/g. High values of iodine number approx. 1300 mg/g, indicated an extensive system of pores and their good adsorption properties.

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INTRODUCTION

Carbon materials have been used by humans since the dawn of time. The first reports of the use of activated carbons (ACs) date back to 1500 BC, where an Egyptian papyrus describes the treatment of rotting wounds with charcoal to remove the unpleasant odor of decaying tissue. Hippocrates and his students used charcoal as a medicine for many diseases. Also, Pliny the Elder, in his work on Natural History, presented coal as a remedy for many ailments, including epilepsy, anemia, and dizziness. Africans and Aborigines, on the other hand, still use charcoal as a poultice to draw poison from wounds inflicted by poisonous animals, including spiders, snakes, scorpions, and others. The Indians, on the other hand, use charcoal to heal burns caused by poison ivy. Currently, AC is used as a remedy for diarrhea and poisoning. On an industrial scale, AC was produced in the first half of the 19th century for the purification of sugar (Bansal and Goyal 2005). At the beginning of the 20th century, activation methods using steam (Ostreijko) or chemical activation (Bayer) methods began to be widely used. During World War I, carbon obtained from coconut shells was used in gas masks. After World War II, the Calgon Corporation USA developed granulated ACs (Dąbrowski 1999).

Activated carbons are currently used in many industries. In recent years, there has been a very rapid increase in both the scale and the range of their various applications.

They are well suited as adsorbents and as catalysts or catalyst carriers (Narowska *et al.* 2019; Abd *et al.* 2021). Because of its ability to store energy, the AC is also used in electrochemistry, *e.g.*, for the construction of capacitors and batteries (Betzy and Soney 2015; Kolonowski *et al.* 2020). The wide use of ACs is owing to their unique porous structure and highly developed specific surface area up to 2000 m^2/g, and the specific chemical structure of their surface.

Currently, the main raw materials used to produce ACs include hard and brown coal, peat, wood, and coconut shells. Obtaining carbon material from non-renewable sources is associated with a negative impact on the environment. In the age of ecology, more and more stringent environmental protection regulations force us to look for new, safe raw materials (precursors) to produce AC.

Great emphasis is placed on the use of lignocellulosic materials, waste from forestry, and agricultural production as well as agri-food production (Danish and Ahmad 2018; Gonzalez-Garcia 2018; Doczekalska *et al.* 2020; Panwar and Pawar 2020). The production of ACs from lignocellulosic biomass has many advantages. Such precursors are diverse, readily available and renewable, cheap, and biocompatible with the natural environment. Moreover, the production of AC from them is a relatively simple process due to the high reactivity of the biomass.

In this study, various anatomical parts of maize were used as precursors. So far, only studies on the preparation of AC from corn cobs have been described in the literature (Aggarwal and Dollimore 1997; Bagheri and Abedi 2009; Kaźmierczak *et al.* 2013; Li *et al.* 2018; Liu *et al.* 2020; Iheanacho *et al.* 2021). Corn (*Zea mays* L.) is one of the staple foods of carbohydrate source substitute for rice. The use of corn has increased tremendously, which causes environmental problems because merely the seeds are used as food while corn cobs and cornstalk waste are discarded and burned. The effort of applying the corn cobs waste as economic material for making activated carbon are beneficial. The structural content of corn cob *i.e.*, cellulose (41%), hemicellulose (36%), and lignin (6%) indicating that corn cob can be potentially and effectively used as a precursor for AC making, besides that the ash content contained in corn cobs is also quite low at 1.50% (Kulp and Ponte 2000). The corn cobs with the high carbon content of 80.5% and low ash content is also an advantage from the other biomass for AC production.

The aim of the study was to obtain and test the properties of ACs from various anatomical parts of maize, *i.e.*, corn kernels, corn silk, corn stalks, corn leaves, and corn cobs without kernels. Potassium hydroxide (KOH) was used as a chemical activator. The properties of the obtained products were evaluated *via* the content of surface oxygen functional groups, parameters of the porous structure, iodine number, and thermal resistance.

EXPERIMENTAL

Materials

The raw material for AC production was common maize (*Zea mays* L.), Legion C1, F1 variety. It was collected in the Wielkopolska-Pomeranian region, Śrem poviat, at its full maturity. Anatomical plant parts were separated from the dried plants (Fig. 1), *i.e.*, corn kernels, corn silk, corn stalks, corn leaves, and corn cobs without kernels which were then crushed into 10-20 mm fractions. The raw materials were stored at a temperature of 20 ± 2 °C, with an RH of 60 ± 5%. The following symbols were used to mark the AC samples obtained from the maize part: AC-K - corn kernels, AC-CS - corn silk, AC-S - corn stalks, AC-L - corn leaves, and AC-C - corn cobs without kernels.

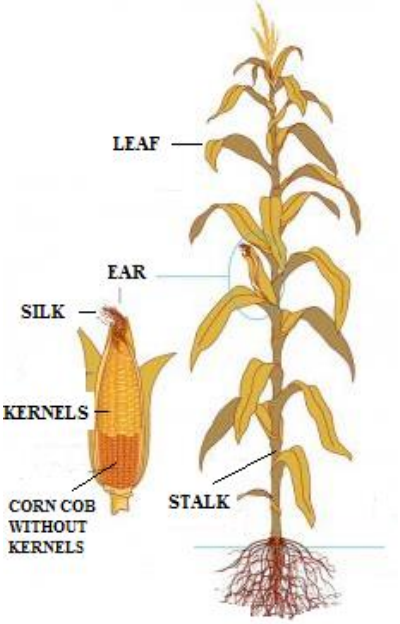

Fig. 1. Parts of corn plants used to obtain activated carbons

Preparation of Activated Carbon

Activated carbons were obtained from various plant parts of corn biomass. Lignocellulosic materials were first subjected to pyrolysis and carbonization in oxygen-free atmosphere by heating to 600 °C at the rate of 3 °C/min and then, holding in stable conditions for 1 h. The carbonized samples after grinding were activated with KOH at mass ratio of 1:4 in argon atmosphere at a temperature of 750 °C for 15 min in a nonporous ceramic reactor. The ACs were washed with 2% hydrochloric acid followed by deionized water to the neutral pH. Prior to use, the ACs were dried in an oven at 105 °C to a constant weight and stored in a desiccator for further study.

Determination of Active Carbon Properties

Surface oxygen groups

This property was determined following Boehm's method (Boehm 1994). Approximately 0.25 g of each AC sample was placed in a 250 mL flask. After adding 25 mL of 0.1 mol/L solution of NaOH, $NaHCO_3$, and 0.05 mol/L solution of Na_2CO_3 (for determination of acidic groups) or 0.1 mol/L HCl (for determination of basic groups), the mixtures were shaken for 24 h. After filtering the mixtures, 10 mL of each filtrate was pipetted and the excess of base and acid was titrated (Tashiro indicator) using 0.1 mol/L solution of HCl or NaOH, respectively. All experiments were repeated twice. The numbers of acidic sites of various types were calculated under the assumption that NaOH neutralizes carboxyl, phenolic, and lactonic groups (Na_2CO_3 - carboxyl and lactonic; and $NaHCO_3$ only carboxyl groups). The number of surface basic sites was calculated from the amount of HCl that reacted with carbon.

Fourier transform-infrared spectra (FTIR)

The AC samples were analyzed on an Alfa FTIR spectrometer (Bruker Optics GmbH, Karlsruhe, Germany) by KBr pellets. The spectra were acquired by accumulating

24 scans at a resolution of 4 cm^{-1} in absorbance mode in the wavenumber range from 4000 to 700 cm^{-1}.

Parameters of the porous structure

The pore parameters of AC were determined from nitrogen sorption isotherms at -196 °C (Micromeritics ASAP 2020, Norcross, GA, USA). Samples before measurement were degassed at 300 °C for 10 h at pressure 1×10^{-6} Pa. Collected sorption data made it possible to calculate the following structural parameters in the area of micro- and mesopores:

- S_{BET} - specific surface area by BET (Brunauer, Emmett and Teller) method - to the relative pressure $p/p_0 \approx 0.2$,
- V_{total} - a total pore volume determined from the isotherm at a relative pressure $p/p_0 \approx 0.975$,
- V_{meso} - mesopores volume by BJH method,
- V_{micro} - micropores volume by t-plot method,
- d_{average} - average pore diameter calculated from formula $d_{\text{average}} = 4V_{\text{total}}/S_{\text{BET}}$.

Iodine numbers (IN)

Iodine numbers of ACs were determined based on the ASTM-D4607-94 standard method. Thus, the IN (mg I_2/g carbon) was measured by titration at 30 °C. From each AC sample, three dried samples (0.1 g each) were placed into separate flasks and fully wetted with 10 mL of 5% HCl. Then 100 mL of 0.025 M standard iodine solution was added into the flask and the content was vigorously shaken for 30 s. After quick filtration, 50 mL of the solution was titrated using 0.1 M sodium thiosulfate using starch as an indicator. The concentration of iodine in the solution was calculated according to Eq. 1 from the total volume of sodium thiosulfate used,

$$\text{IN} = \frac{(V_0 - V_x)\times c_{thio}\times 126.92}{m}\ [\frac{mg}{g}] \tag{1}$$

where V_0 and V_x are the volumes of sodium thiosulfate solution used for titration of assayed (V_x) and blank samples (V_0) [mL]; c_{thio} is the concentration of sodium thiosulfate solution [M]; m is the mass of activated carbon sample [g]; and 126.92 is the mass of 1 mole of iodine [g].

Thermogravimetric analysis (TG)

The analysis was carried out on STA 449 F5 Jupiter-QMS of the NETZSCH (MA, USA) using the following conditions: final temperature 1200 °C, temperature increase at 5°C/min, and helium flowing at the rate of about 25mL/min. The weight of the sample used was 10 mg ±1 mg.

RESULTS AND DISCUSSION

The porous structure of ACs, and thus their adsorption capacity, is largely determined by the properties of the starting material and the conditions used for its preparation, such as carbonization parameters (*i.e.*, end temperature, cooling method and rate, temperature raising rate, and atmosphere) and the type of the factors used in the activation process.

Table 1 summarizes different preparation methods of corn cobs based ACs (AC-C sample) and their BET surface area (S_{BET}). Comparing the various methods used to obtain ACs from corn cobs, it can be stated that the specific surface area obtained by chemical methods was much larger than that obtained by physical methods. Based on these reports, in this work, the ACs were obtained by chemical activation method, and KOH was used as an activator.

Table 1. Comparison of ACs from Corn Cob with Different Activation Methods

Preparation Method	BET Surface Area (m^2/g)	Reference
Chemical, KOH	1682	Tsai *et al.* 2001
Chemical, KOH	1600	Song *et al.* 2013
Chemical, KOH	1320	Bagheri and Abedi 2011
Chemical, KOH	1054	Liu *et al.* 2020
Chemical, KOH	1884	This study
Chemical, K_2CO_3	1266	Tsai *et al.* 2001
Chemical, $ZnCl_2$	924.9	Tang *et al.* 2016
Physical, Steam	980	Song *et al.* 2013
Physical, Steam	630	Ioannidou *et al.* 2010
Physical, CO_2	431	Milenković *et al.* 2013
Physical, CO_2	143	Nethaji *et al.* 2013

The results from other reported studies (Lillo-Rodenas *et al.* 2003, Linares-Solano *et al.* 2008, Raymundo-Piñero *et al.* 2005) indicate that carbon hydroxide activation occurs through the reaction of Eq. 2.

$$6\ MOH + 2\ C \leftrightarrow 2\ M + 3\ H_2 + 2\ M_2CO_3 \qquad (2)$$

(where M is Na or K)

This solid-liquid redox reaction transforms hydroxides, a result of oxidation of the carbon precursor, into the main products: hydrogen, alkali metals, and alkali carbonates. Often this reaction (Eq. 2) is carried out at a temperature range of 700 °C to 900 °C.

Two types of functional groups determine the chemical structure of the surface of ACs (Bansal and Goyal 2005). Acidic functional groups such as carboxyl, phenolic, carbonyl, lactone, anhydride groups are very well characterized and give the carbon structure a hydrophilic and polar character. The structure of alkaline functional groups of pyronium type structure is not fully understood, but they are also classified as chromene structures. This structure has an oxygen-containing heterocyclic ring fused to an active group of $=CH_2$ or $=CHR$ (where R is an alkyl group).

The presence and type of oxygen functional groups present on AC surface primarily determine the ion-exchange properties of AC. They also affect their adsorption, electrochemical, catalytic, redox, and hydrophobic-hydrophilic properties. The determination of the content of acidic and alkaline oxygen functional groups on the surface of AC was carried out using the Boehm method and is summarized in Table 2.

There was a substantial amount of surface oxygen groups on the surface of the ACs from corn biomass (Table 2). Activated carbons showed an acidic surface character. In the case of carbon material obtained from AC-L, the advantage of acidic groups over basic groups was small. The results of the determinations indicate that carboxyl groups provided the greatest share in acidity. The low content of lactone groups is most likely due to their inferior thermal resistance compared to phenolic and carboxyl groups (Szymański *et al.*

2002). On the other hand, the highest number of phenolic groups was found in the case of carbon produced from AC-S.

Table 2. Surface Oxygen Functional Groups

ACs	Functional Groups (mmol/g)				
	Acidic			Acidic (Total)	Basic (Total)
	Lactonic	Phenolic	Carboxylic		
AC-K	0.29	0.63	1.23	2.15	0.77
AC-CS	0.30	0.62	1.33	2.25	0.48
AC-S	0.20	0.77	1.26	2.23	0.53
AC-L	0.15	0.62	1.27	1.93	1.83
AC-C	0.29	0.58	1.27	2.14	0.68

AC samples obtained from the maize part: AC-K - corn kernels, AC-CS - corn silk, AC-S - corn stalks, AC-L - corn leaves, and AC-C - corn cobs without kernels

In the FTIR spectra of ACs (Fig. 2), the absorption bands at similar wavenumbers in the vibrational region of 4000 to 2000 cm^{-1} are visible. The most intense bands were apparent around 3430 to 3420 cm^{-1}, and they come from the stretching vibrations of –OH groups in various configurations and also from chemisorbed water. The absorbance bands at 2920 cm^{-1} and 2850 cm^{-1} are characteristics of -C-H stretching vibrations in aromatic and aliphatic structures and for the $-CH_3$ and $-CH_2$ groups. The peaks in the range 1740 to 1730 cm^{-1} on the carbon spectra can be attributed to the stretching vibrations of the –C=O bonds from carboxyl, ester, and lactone groups.

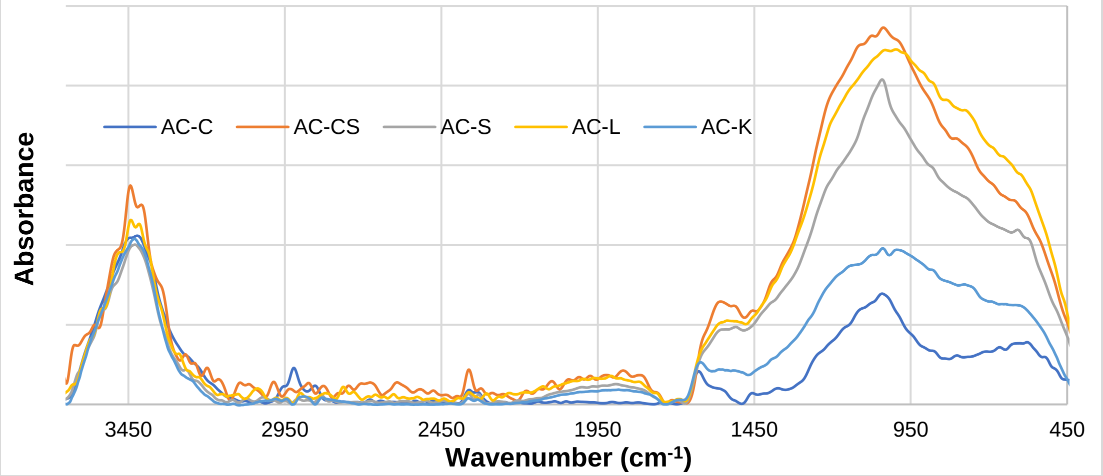

Fig. 2. Infrared spectra of activated carbon (see ACs abbreviations below Table 2)

Boehm's method showed the presence of carbonyl groups, which is confirmed by the presence of the band at the wavenumber of 1740 cm^{-1}. Additionally, the spectrum of the carbon showed a band of vibrations at 1635 cm^{-1}, corresponding to the stretching vibrations of -C=O bond. On the other hand, the spectra show a band at 1558 cm^{-1}, which arises from the -C=O stretching vibrations of diketones, ketoesters, and ketoenol groups or the -C=C stretching vibrations of aromatic rings (Biniak *et al.* 1997; Figueiredo *et al.* 1999;

Pietrzak and Wachowska 2003; Pietrzak and Bandosz 2007). The similarity of the spectra was found in the case of corn kernels and corn cob without kernels. In their case, the absorbance was the lowest, and there was no evidence of any band below 1537 cm^{-1}, which was present in the spectra of other ACs samples.

Results of the Boehm's method (Table 2) and FT-IR measurements made for the ACs are shown in Fig. 3. The table provides information about the functional groups formed on the material surface, which are very important for the evaluation of the adsorption properties.

Textural parameters are considered to have a strong influence on the adsorption efficiency of an AC sample. Hence, textural characterizations were performed (Table 3) to determine the surface area (S_{BET}) as well as the micropore (V_{micro}) and mesopore (V_{meso}) volumes of the adsorbents. The nitrogen adsorption-desorption isotherms are presented in Fig. 3.

Table 3. Characteristic of the Porous Structure of the Activated Carbon

ACs	Surface Area (m^2/g)	Pore Volume (cm^3/g)			V_{micro}/V_{total}	Pore Width (nm)	Iodine Number (mg/g)
	S_{BET}	V_T	V_{micro}	V_{meso}		$d_{average}$	*IN*
AC-K	1965	1.12	0.32	0.80	0.29	2.28	1280
AC-CS	1600	1.15	0.53	0.62	0.46	2.88	1290
AC-S	1948	1.27	0.33	0.94	0.26	2.61	1290
AC-L	1721	1.30	0.67	0.63	0.51	3.02	1280
AC-C	1884	1.19	0.31	0.88	0.26	2.53	1310

Note: See ACs abbreviations below Table 2.

For almost all isotherms (Fig. 3), quite high adsorption values can be found in the range of low relative pressures (up to 0.1), *i.e.*, about 500 cm^3/g. This may indicate the presence of a micropore system. These curves are also characterized by steep courses in the range of relative pressures from 0.1 to 0.4, in which nitrogen is absorbed mainly in the mesopores. High adsorption values (over 800 cm^3/g) are characteristic of carbons obtained from corn stalks (AC-S) and leaves (AC-L).

All the obtained activated carbons were characterized by a high specific surface area, *i.e.*, from 1721 m^2/g to 1965 m^2/g, and a high total pore volume between 1.12 cm^3/g and 1.30 cm^3/g. The largest S_{BET} areas were obtained by using corn kernels and stalks as precursors. Surface area (S_{BET}) values increased in the order: AC-CS < AC-L < AC-C < AC-S < AC-K. The V_{micro} values were significantly lower than V_{meso} for almost AC samples. The exception was AC-L. In this case, the ratio V_{micro}/V_{total} was 0.51. It was also characterized by the highest average pore diameter, *i.e.*, 3.02 nm. The volume of micropores ranged from 0.31 cm^3/g (AC-C) to 0.67 cm^3/g (AC-L).

The IN is widely used as a quality control parameter in the production and regeneration of AC. Iodine adsorption is considered as a simple and quick test for evaluating the porous structure. Iodine has a small molecular size and it can readily penetrate deep micropores of the AC. So, the IN values gives approximate measure of the micropore content of the carbon. Based on the obtained results (Table 3), it was found that all carbons were characterized by a high iodine adsorption capacity - approx. 1300 mg/g.

These high iodine values confirm their developed porous structure and indicate a good adsorption capacity. Determining the adsorption properties should be the subject of the next stage of research. At present, it can be concluded that for all ACs, a large specific surface area and a large pore volume were found.

The prepared ACs were also tested using the thermogravimetric method in the temperature range of 35 °C to 1200 °C (Table 4).

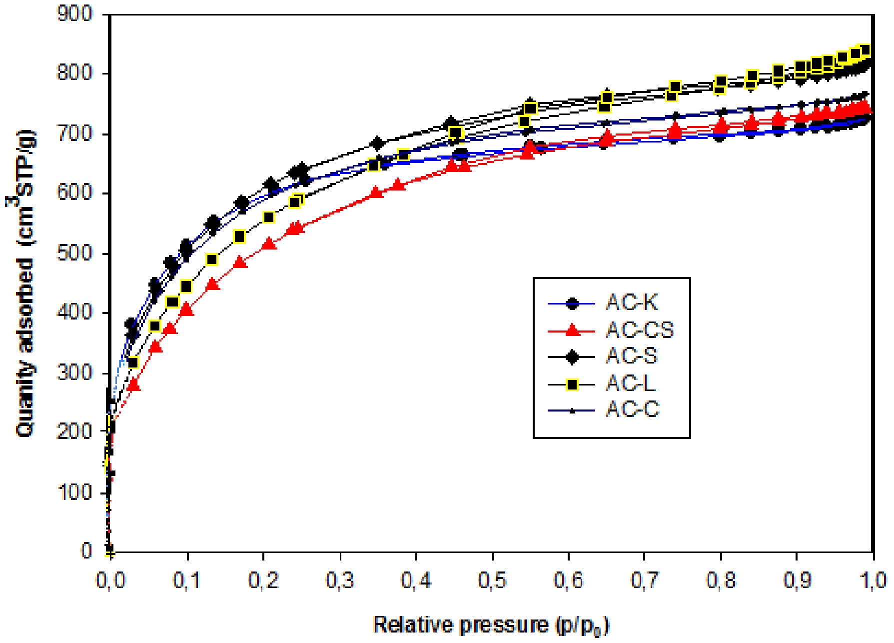

Fig. 3. N_2 adsorption-desorption isotherms of activated carbon (see ACs abbreviations below Table 2)

Table 4. Thermogravimetric Analysis of ACs

ACs	Weight Loss (%)					
	35-200 °C	200-500 °C	500-700 °C	700-900 °C	900-1200 °C	35-1200 °C
AC-K	3.17	5.77	7.71	7.29	9.20	33.14
AC-CS	0.86	2.97	3.97	5.48	6.88	20.16
AC-S	1.92	5.87	6.82	6.43	7.87	28.91
AC-L	1.49	6.23	6.50	5.98	8.14	28.34
AC-C	2.34	4.73	6.51	6.25	6.87	26.70

Note: See ACs abbreviations below Table 2.

During thermogravimetric tests, thermal decomposition of surface oxygen groups was observed. They exhibited different thermal stabilities because they are formed in places with different energies. The weight loss in the temperature range from 35 °C to 200 °C was mainly related to the evaporation of water present in the sample, and the greatest changes in this range were recorded for AC-K. In the temperature ranges of 200 to 500 °C and 500 to 700 °C, the thermal decomposition of surface acid oxygen groups occurs. Decomposition of basic oxygen groups is observed at higher temperatures (Boehm 2002). The largest loss of mass in the entire temperature range was exhibited by AC-K. On the other hand, the lowest total weight loss in the range of 35 to 1200 °C (20.16%), as well as in each temperature range, was recorded for AC-CS.

CONCLUSIONS

1. Various anatomical parts of maize used in the study turned out to be good raw materials to produce activated carbon. All the obtained ACs were characterized by a rich chemical surface structure, well-developed porous structure, and sufficient thermal stability.
2. The high content of surface acid oxygen groups (approx. 2 mg/g) in the prepared ACS were confirmed by Boehm methods and infrared spectroscopy. All ACs have an acidic surface character with the highest proportion of carboxyl groups.
3. The obtained activated carbons are characterized by a highly developed specific surface ranging from 1600 to 1965 m^2/g. The largest S_{BET} areas were obtained by using corn kernels and stalks as precursors.
4. Activated carbons from various anatomical parts of maize are also characterized by a high proportion of mesopores in the internal capillary structure. The IN from all ACs were about 1300 mg/g, which may indicate an extensive pore system and their good adsorption properties.

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