

Effect of treatment on the production and characterization of activated carbon from soya bean husk

Abstract

This study investigates the physicochemical transformation of soybean husk into activated carbon using phosphoric acid (H_3PO_4) and potassium carbonate (K_2CO_3) as activating agents. Results revealed significant reductions in moisture content from 10 % in the raw husk to 5 % and 3 % in acid and base-activated samples, respectively, indicating dehydration and increased hydrophobicity. Ash content increased in both treatments more markedly in the base-activated carbon due to retention of potassium salts and other inorganic residues. Bulk density rose from 0.38 g cm^{-3} in the raw husk to 0.54 g cm^{-3} (acid) and 0.62 g cm^{-3} (base), suggesting improved structural consolidation. Surface pH shifted from 6 to 7.4 and 8.0 for acid- and base-activated carbons, respectively, reflecting modified surface acid-base behavior and adsorption selectivity. FTIR analysis confirmed the removal of O–H, C=O, and C–O groups, enhancement of aromatic C=C bonds, and emergence of P–O and K–O vibrations associated with the activating agents. Overall, both activation routes successfully produced porous, functional carbon materials. H_3PO_4 activation favored microporous, phosphate-enriched carbon suitable for neutral or organic pollutant adsorption, while K_2CO_3 activation generated hydrophobic, alkaline carbon advantageous for acidic species removal and catalytic applications.

Keywords: soya bean husk, fourier transform infrared spectroscopy, acid treatment, base treatment, bulk density, potential of Hydrogen

Introduction

Activated carbon is a remarkable adsorbent for a variety of substances because of its large internal surface area and porous nature.¹ As a multipurpose adsorbent, it finds use in a variety of industrial fields, including chemical processing,² air filtration,³ and water purification.⁴ The activation process, which includes carbonization and subsequent physical or chemical treatment to create a porous structure, is what gives it its special qualities.⁵ The economic viability⁶ and favorable environmental impact⁷ of producing activated carbon from easily accessible agricultural by-products have attracted a lot of research attention recently.

Activated carbon has been utilized for medicinal and water purification purposes since ancient Egypt.⁸ Its uses today are numerous and include: Treating water and wastewater by eliminating organic pollutants, taste-or odor-causing substances, and impurities.⁴ Reducing smell, eliminating volatile organic compounds (VOCs), and cleaning industrial gases are all ways to purify the air.³ Recovery of gold through extraction from solutions.⁹ Medical uses include wound care, detoxification, and medication delivery.¹⁰ For decolorization, purification, and odor elimination in the food and beverage industry.¹¹ About 10–15% of the total weight of soybeans is made up of soybean husk, a waste product produced during the processing of soybeans.¹² It is a desirable precursor for the production of activated carbon due to its properties: High carbon content (40–50%),⁶ Low cost and abundant availability,⁷ and environmentally friendly (reduces waste disposal issues).¹²

This study focuses on the use of soybean husk as a low-cost activated carbon for commercial purposes using phosphoric acid (H_3PO_4) and potassium carbonate (K_2CO_3) as activating agents.

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Materials and methodology

Materials

Waste soya bean husk was obtained from Samaru, Zaria. The soya bean husk was crushed, ground, and sieved to obtain a fraction having particle sizes of 1.7–2.8 mm.

Preparation of activated carbons

To lower the moisture content, soy bean husk was first sun-dried and then baked at 100 degrees Celsius for 24 hours. In order to create a carbon-rich substance, the dried sample was carbonized for two hours at 400 degrees Celsius in a furnace without oxygen. The carbonized material was activated using a 0.1M activating agent, such as potassium carbonate (K_2CO_3) and phosphoric acid (H_3PO_4) in a ratio of 1 to 4, or soy husk to solution. After two hours of heating at 400 degrees Celsius, the activated material should be filtered and rinsed with distilled water to get rid of any contaminants. After two hours of oven drying at 100 to 150 degrees Celsius, the activated carbon was ground into a fine powder.

Proximate analysis

Proximate analysis, as defined by ASTM D 121, is the process of determining moisture, ash content, pH, and bulk density using specified techniques. The following process was used to perform the proximate analysis of the various types of activated carbon.

Moisture content

After measuring the weight of a small sample of activated carbon (soy shell), it was placed in a petri dish. It was evenly distributed across the plate. After that, it was baked for 1.5 hours at a temperature

between 105 and 110 degrees Celsius. During the heating process, the petri dish was either left open or uncovered. Following heating, the petri dish was taken out and allowed to cool in a desiccator. The dried sample's weight was determined once it had cooled.

$$\text{Moisture content } M = 100(B-F)/(B-G)$$

B=weight of petri dish +original sample

F=weight of petri dish+ dried sample

G= weight of petri dish

Ash content

A silica crucible was filled with one gram of the sample. It was heated for one and a half hours to 750°C in a muffle furnace. The crucible was left open throughout this heating process. The crucible was cooled in a desiccator following the necessary heating, and the weight of the ash was then determined.

$$\text{Ash content } A = 100(F-G)/(B-G)$$

G=Mass of empty crucible

B=Mass of crucible + sample

F=Mass of crucible+ ash sample

Bulk density

The weight of a material per unit volume is known as its bulk density. Basically, it is applied to powdered materials. The bulk density test indicates the solid sample's packaging quantity and flow consistency. The unit of measurement is kilograms per cubic meter (kg/m³). The mass of the measuring cylinder used in this experiment should be determined first. After that, the provided activated carbon sample was put inside the cylinder and weighed again. After that, it was placed on the aluminum plate and oven-dried for 60 minutes at 105°C. The dry sample's weight was determined after it had dried.

$$DB = (m_2 - m_1)/v$$

M1 mass of measuring cylinder in grams

M2 mass of measuring cylinder + its contents

V volume of the measuring cylinder in litres

pH

pH was determined using the standard method ASTM D 3838-80. 1g of activated carbon of

The soya shell sample was put in a conical flask, and 100ml distilled water was added to it.

The mixture was stirred for 1hour. pH readings were taken using a pH meter. Repeat the

Procedure for the other two types of activated carbon.

Fourier transformed infra-red spectroscopy (FTIR)

Fourier transform infrared spectroscopy was used to determine the surface functional groups

groups of the activated carbon obtained from soya bean husk.

Results and discussion

After the production process, the result showed that the AC obtained from the base-activated carbonized husk was darker and retained more water than the acid-activated carbonized husk. Figure

1, Compared with the untreated soybean husk, the moisture content is significantly reduced by both acid and base treatments. This is expected since carbonization eliminates volatile substances, organic matter, and functional groups that aid moisture. Because hydroxyl and other polar groups are lost, the carbonized husks are typically more hydrophobic. The moisture content of the base-activated carbonized husk is marginally lower than that of the acid-activated husk as reported by Togibasa et al.¹³ Base activation (potassium carbonate) produces a more non-polar and hydrophobic surface, which further reduces moisture uptake, acid activation (phosphoric acid) tends to introduce more oxygen-containing groups, which may still retain some moisture (Figure 1).

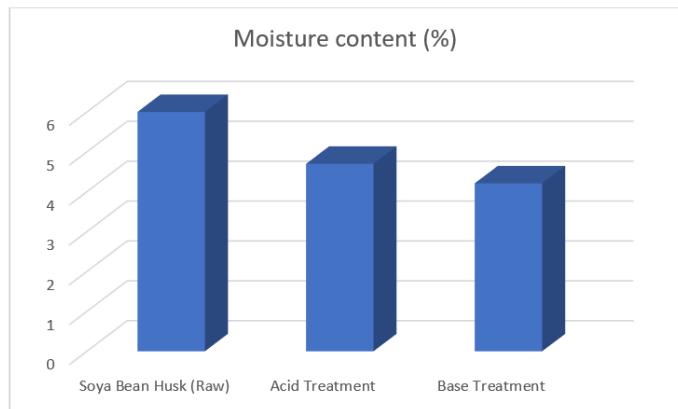


Figure 1 Moisture content of raw soya husk, acid treatment, base treatment

Ash content (%)

The amount of inorganic residue that remains after full combustion, which usually consists of minerals like silica, potassium, calcium, and magnesium, is measured by the ash content of materials like soybean husk and its carbonized forms (treated with acid and base). Since it influences the material's potential for use in processes like adsorption, catalysis, and filtration, the amount of ash is a crucial indicator of the material's purity and degree of activation, particularly when working with biochars or activated carbons (Figure 2).¹⁴

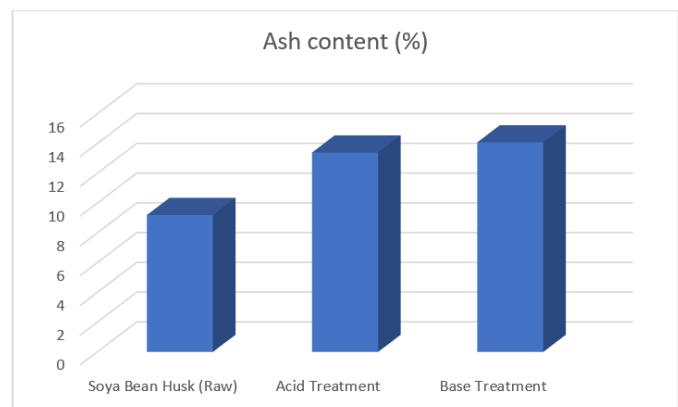


Figure 2 Ash content of raw soya husk, acid treatment, base treatment

Both base and acid activation raise the ash content, with base treatment producing the highest ash value. Depending on the type of inorganic components (phosphates or potassium compounds), a higher ash content can improve the material's application for particular uses, like catalysis or ion exchange, but it can also decrease the overall carbon yield and adsorption potential because the ash represents non-carbon residues. Since a high ash content may decrease the available surface area, a lower ash content in raw or mildly carbonized material

is preferable for adsorption applications (such as the removal of dyes or pollutants). However, because of their increased chemical reactivity, phosphates or potassium residues may be advantageous for catalysis or specific specialized environmental applications.¹⁴

Bulk density

The mass of a material, including its solid components as well as any void spaces, per unit volume is referred to as its bulk density. Because it influences the packing density, porosity, and overall material performance, it is a crucial characteristic when assessing materials for a variety of applications, especially in adsorption, filtration, and catalysis as reported by Szymańska & Strzelczyk (Figure 3).¹⁵

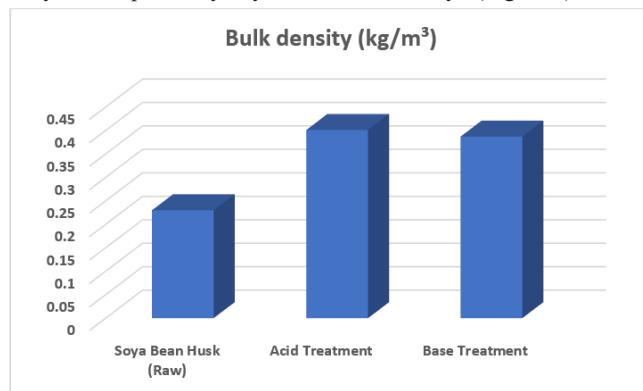


Figure 3 Bulk density of raw soya husk, acid treatment, base treatment

Since phosphoric acid usually produces more micropores and condensed structures, whereas potassium carbonate produces a more open structure with macropores, resulting in a slightly lower bulk density, the higher bulk density of acid-activated carbon suggests it may be more suitable for applications requiring a higher surface area with compact packing, such as in adsorption or catalysis.¹⁶ The increase in bulk density following chemical activation is expected and indicates successful carbonization and activation, which removes volatile compounds and reorganizes the carbon structure. With a somewhat lower bulk density, base-activated carbon might be more appropriate for uses requiring lighter, more porous materials, like water filtration or as lightweight adsorbents. Thus, depending on its bulk density and pore structure both of which are impacted by the activation technique employed each type of carbonized soy bean husk can be customized for particular uses.

pH

Because soy bean husk contains naturally occurring organic acids like phenolic acids and carboxyl groups from cellulose, hemicellulose, and lignin, it usually has a slightly acidic nature (pH ~6). The pH of the substance is typically lowered by these organic acids. Because of its organic makeup, pH 6 is naturally slightly acidic, which restricts its adsorption potential in the absence of additional treatment. A more balanced surface chemistry is produced by carbonization with acid treatment (pH 7.4), which makes the material appropriate for a variety of uses, particularly in adsorption processes where neutral pH is advantageous, as reported by Rahman et al.¹⁷ An alkaline character is imparted by carbonization with base treatment (pH 8), which enhances the material's capacity to adsorb acidic species Hupian et al.¹⁸

This is advantageous in environmental applications where the elimination of acidic pollutants is required. Each pH level is therefore appropriate for a distinct industrial or environmental use, reflecting how chemical treatments (acid vs. base) change the surface chemistry and possible uses of carbonized soy bean husk (Figure 4).

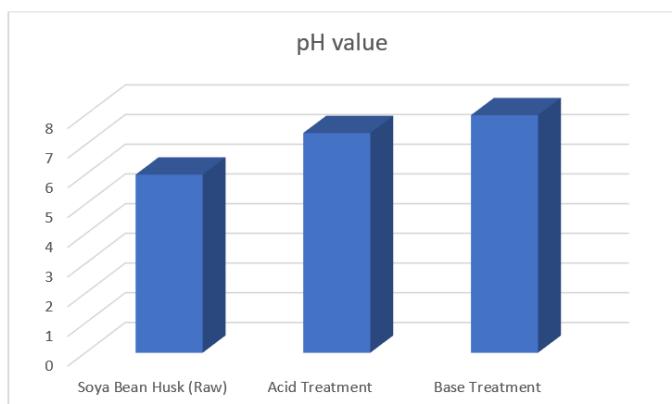


Figure 4 pH value of raw soya husk, acid treatment, base treatment

The highest moisture content (10%) is found in soybean husk, which drastically drops in both carbonized forms (3% for base treatment and 5% for acid treatment). Carbonization and treatment increase bulk density, which is 0.38 g/cm³ for raw husk, 0.54 g/cm³ for acid treatment, and 0.62 g/cm³ for base treatment. Following carbonization and chemical activation, this increase suggests a denser material. After being treated with acid, the pH rises from 6 in the raw husk to 7.4, indicating a more neutral pH. After being treated with base, the pH further rises to 8, indicating a basic environment.

Fourier transform infrared spectroscopy (FTIR) analysis

The FTIR spectrum for **soya bean husk** and carbonized husk is shown in the Figure below characteristic peaks that provide insights into the molecular structure of the material. The spectra show reduction of O–H stretching (~3273 cm⁻¹) and C=O bands (~1739 cm⁻¹), indicating dehydration and decarboxylation during carbonization/activation; enhancement of aromatic C=C features (~1608 cm⁻¹), consistent with lignin condensation and increased aromaticity; reduction of C–O/polysaccharide bands (~1014 cm⁻¹) consistent with cellulose/hemicellulose degradation; and new bands in treated samples attributable to K–O/carbonate species for base activation and P–O/phosphate bands for acid activation. These spectral changes confirm that activation both removes oxygenated oxygen-rich functionalities and introduces inorganic residues characteristic of the activating agent, which will in turn control hydrophilicity and reactivity (Figure 5).

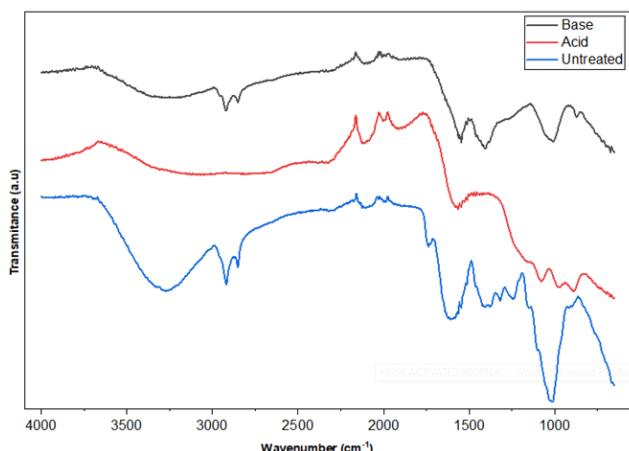


Figure 5 FTIR spectra of untreated soya bean husk, carbonized soya bean husk activated with phosphoric acid (H_3PO_4), and carbonized soya bean husk activated with potassium carbonate (K_2CO_3).

The FTIR spectrum of untreated and carbonized soya bean husk displays several key functional groups and transformations,

providing insights into the chemical structure changes that occurred due to the treatment, as presented in (Table 1).

Table I The summary of changes in FTIR spectra that includes both the base treatment (potassium carbonate) and acid treatment (phosphoric acid) of soya bean husk

Functional group	Untreated husk	Treated husk (base activated)	Treated husk (acid activated)
O-H stretching (~3272.8 cm ⁻¹)	Strong peak, indicating high hydroxyl content.	Reduced intensity, showing partial dehydration and structural changes.	Reduced intensity, indicating acid-catalyzed dehydration and breaking of hydroxyl groups. ^{19,20}
C-H stretching (2922.2 & 2851.4 cm ⁻¹)	Strong, indicating the presence of aliphatic chains.	Present but weakened, showing partial degradation of hydrocarbons.	Slightly reduced, suggesting dehydration and partial breakdown of hydrocarbons. ^{20,21}
C=O stretching (1738.9 cm ⁻¹)	Pronounced, suggesting high carbonyl content.	Reduced or shifted, indicating decarboxylation during activation.	Significantly reduced, indicating removal of carbonyl groups, possibly due to phosphoric acid oxidation. ^{23,24}
Aromatic C=C (1608.5 cm ⁻¹)	Less prominent, indicating lower aromaticity.	Enhanced, showing increased aromatic content due to lignin condensation.	Similar enhancement due to increased aromaticity from acid treatment and lignin restructuring. ^{25,26}
C-O Stretching (~1013.8 cm ⁻¹)	Strong, indicating high polysaccharide content.	Reduced intensity, indicating partial degradation of polysaccharides.	Significant reduction, showing degradation of cellulose and hemicellulose during acid treatment. ^{7,27}
New peaks (~670.9 cm ⁻¹ in base treatment)	Absent.	New peak indicates K-O bonds or carbonate residues introduced by base treatment.	New peaks associated with P-O and phosphate-related functional groups from phosphoric acid. ^{7,23}

From the table both base and acid treatments cause structural modifications in the soya bean husk, as observed through FTIR spectra. Base treatment enhances the aromatic nature and introduces carbonate-based structures, while acid treatment significantly affects cellulose and carbonyl groups and incorporates phosphate functionalities. The choice of activation method will influence the husk's final properties, making the carbonized material suitable for different applications, such as adsorption, based on the desired surface chemistry.

Conclusion

Both acid (H_3PO_4) and base (K_2CO_3) activation effectively enhanced the physicochemical properties of soybean husk-derived carbon through dehydration, structural reorganization, and surface modification. Moisture content decreased from about 10 % in the raw husk to 5 % and 3 % in acid- and base-activated samples, respectively, and increased hydrophobicity. Ash content rose in both treatments, with the base-activated carbon exhibiting the highest value due to residual potassium salts. Bulk density increased from 0.38 g cm^{-3} (raw) to 0.54 g cm^{-3} (acid) and 0.62 g cm^{-3} (base), suggesting formation of a denser structure and possible inorganic residue retention. The surface pH shift from 6 in the raw husk to 7.4 (acid) and 8 (base) shows that activation tailors surface acidity and adsorption selectivity, with base-treated carbon favoring acidic species and acid-treated carbon more suited for neutral organics. FTIR spectra confirmed loss of O-H and C=O groups, enhancement of aromatic C=C bands, reduction of C-O polysaccharide signals, and appearance of P-O or K-O vibrations, indicating lignin condensation and incorporation of activating-agent residues. Overall, both activation routes converted soybean husk into functional carbon materials with distinct surface chemistries. Acid activation produces microporous, phosphate-rich carbon ideal for organic pollutant adsorption, and base activation yields hydrophobic, alkaline carbon better suited for acidic species removal or catalytic applications.

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Conflicts of interest

The author declares that there is no conflicts of interest.

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