Activation And Analysis of Tyre Char Obtained from Waste Tyre Pyrolysis

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***Abstract*—The development of better and efficient methods of consuming less and/or wasting little resource materials is becoming more important. In this study, Thermal pyrolysis of waste tires has been studied in a rotating kiln reactor under nitrogen atmosphere. The pyrolysis temperature was varied in a wide range to investigate the effect of temperature on pyrolysis products. The pyrolytic char during each pyrolysis process was reported in different pyrolysis times. However, by raising the temperature of the pyrolysis process, the yield of produced char decreased. Pyrolytic waste tyre carbon black residue and commercial grade activated carbon were characterized and evaluated.**

***Index Terms*—Waste tire, Pyrolysis, Rotary kiln reactor, Sulphuric Acid, Hydrogen Peroxide.**

I. INTRODUCTION

Disposal of used tires is a critical environmental waste management issue over the world. Every year, enormous amount of waste tires is generated by increasing the transportation demand. It is estimated that about 1.5 billion waste tyres are produced annually worldwide Tires are usually made of widely different rubbers, carbon black, and small amount of organic and inorganic additives. The complex and cross-linked structures of rubbers cause tires to become highly resistant to photochemical decomposition, biodegradation, chemical reagents, and high temperatures. The economics of the pyrolysis plant is very sensitive to the yields of all products, and generally, the pyrolysis process is integrated with other processes for obtaining higher value-added products. Therefore, management of waste tires is a serious technological, economic and ecological challenge Feasible way for recovering valuable hydrocarbons from waste tires and obtaining chemical feedstock and energy is an interesting subject for researchers. Pyrolysis is a process in which organic materials are thermally decomposed when subjected to an elevated temperature in the absence or in the presence of very low account of oxygen. Thus, many attempts have been made to investigate recycling of waste tires by pyrolysis process.

The most significant part of pyrolysis process is furnaces (reaction chamber) and the heat transfer system because of low thermal conductivity of tires. With development of technology, a wide range of dissimilar reactors such as pressurized or vacuum, continuous or batch, catalytic or non-catalytic and fluidized or fixed bed was investigated by various researchers to enhance pyrolysis performance. In many pyrolysis experiments, heat is provided by an external source to the reactor such as electrical resistance or fuels combustion. The main parameters investigated in pyrolysis studies are tire size, reactor configuration, temperature, pressure, and residence time. Because of the reliability of thermogravimetric systems, they are usually the first choice in tire pyrolysis studies. These reactors are usually used to estimate the kinetic parameters of tire decomposition. Fixed bed reactors can be easily constructed, and they are commonly used in laboratory experimental studies of waste tires studied the pyrolysis powder of scrap truck tire in a fixed bed reactor and reported that the higher heating rates promote the gas production at the expense of oils. pyrolyzed tire wastes in a fixed-bed fire-tube heating reactor under different pyrolysis conditions to determine the effects of final temperature, sweeping gas flow rate, and feed size on product yields and compositions. The final temperature range for pyrolysis studies was between 375 and 575 C, and the highest liquid product yield was obtained at 475 C. introduced pyrolysis at the temperature of 430 C, nitrogen flow rate of 0.35 m3 h1 and particle size of 10 mm as the optimum condition in which the maximum yields were reported. At these conditions, the yields of carbon black and pyrolytic oil were 32.5 and 51.0 wt%, respectively. found that by pyrolysis of a truck tire, more amount of oil and less amount of carbon black were obtained comparing to that of a passenger car tire. studied non-catalytic and catalytic pyrolysis experiments of tire in a stainless steel fixed-bed reactor and reported that by increasing the catalyst-to-tire ratio, the yield of pyrolytic oil increased and reached a maximum of 65.11 wt% at the ratio of 0.10. This result was 8.48 wt% higher than that of non-catalytic pyrolysis.

Inspite of difficult operating conditions of fluidized bed reactors, these reactors have particularly high heat transfer. Effects of different parameters such as particle size, feed rate, and pyrolysis temperature on pyrolysis process were investigated in fluidized beds. A circulating fluidized bed for waste tire pyrolysis was developed. They found that a long residence time contributes to secondary reactions, lower temperature and heating rate, in favor of carbonization which reduces the oil yield. The pyrolysis of shredded tire wastes at the atmospheric pressure under inert gas atmosphere in a fluidized bed combustion setup. They found that a low pyrolysis temperature of 400 C with low feed rate, increases residence time in the combustion reactor yielding maximum oil. Also, they reported that interaction between temperature and feed rate is important in deciding the oil yield; since they control residence time and temperature of feed stock inside the reactor. The scrap tire pyrolysis in a continuous two-stage pyrolyzer consisting of an auger reactor and fluidized bed reactor and various additives and fluidizing media. They reported that N2 reduced the sulfur content in pyrolysis oil more effective than product gas, due to its dilution effect. Carried out continuous pyrolysis of scrap tire in a conical spouted bed. The main difference between the continuous and batch processes is the yield of light aromatics, which is higher in a continuous process. However, for heavy liquid fraction or tar, the yield of light aromatics is higher in a batch process. The pyrolysis of waste tires integrated with concentrated solar power using linear Fresnel reflectors technology.

In the current work, pyrolysis process including a laboratory scale rotary kiln was designed for waste tire pyrolysis by thermal degradation. As temperature is the significant parameter affecting pyrolysis yields, a wide range of pyrolysis temperature from 400 to 1050 C was investigated, and the reactor was heated in two sections (temperature raising and temperature constant sections). The yields of pyrolytic oil, gas, and char at various temperatures were evaluated in order to identify maximum produced pyrolytic oil. Moreover, by using FT-IR analysis, functional groups and types of bonds at different temperatures were determined. Finally, the properties of obtaining maximum pyrolytic oil such as distillation were analyzed.(1-2)

1.1 ROTARY KILN REACTOR

A rotary kiln reactor is an essential technology used in the pyrolysis of waste tyres, offering an efficient and sustainable method for converting waste into valuable products. Pyrolysis is a thermal decomposition process carried out in the absence of oxygen, where tyres are subjected to high temperatures, typically between 400°C and 700°C, inside the rotating kiln. The rotary kiln reactor ensures continuous and controlled processing of tyre material, which leads to the breakdown of the complex hydrocarbons in the rubber into simpler compounds, producing pyrolysis oil, syngas, and carbon black. This technology has gained widespread application due to its ability to reduce environmental pollution and recover valuable resources from waste tyres.

The design of a rotary kiln for tyre pyrolysis is optimized to handle the specific characteristics of rubber materials. The kiln is a long, slightly inclined cylindrical reactor, with tyre feedstock introduced at one end and processed material exiting at the other. The slow rotation of the kiln ensures that the tyre materials move gradually through the reactor, allowing for uniform heat distribution and efficient pyrolysis. The internal temperature is carefully controlled through burners or external heating systems, and the rotary motion helps prevent the material from agglomerating or sticking to the walls, thus facilitating continuous operation.

During the pyrolysis process in the rotary kiln, waste tyres are decomposed into three main products: pyrolysis oil, carbon black, and syngas. Pyrolysis oil is a valuable liquid that can be further refined into fuels like diesel or used directly in some industrial applications as a heating source. Carbon black is a solid residue that can be used in rubber reinforcement, pigments, or as an energy source. Syngas, a mixture of combustible gases, can be utilized to power the pyrolysis system itself or for other energy applications, improving the overall energy efficiency of the process. A significant advantage of using a rotary kiln reactor for tyre pyrolysis is its ability to operate continuously, processing large volumes of waste tyres efficiently. The rotary design allows for steady feedstock movement and mixing, resulting in consistent product quality and efficient heat transfer. The pyrolysis gases generated during the process can be recovered and reused as fuel, reducing the overall energy consumption and making the process more sustainable. Additionally, rotary kilns are highly adaptable and can be designed to accommodate different feed rates, sizes of tyres, and operational conditions, making them suitable for both small-scale and large-scale operations.

Furthermore, the pyrolysis process in rotary kilns offers an environmentally friendly solution for waste tyre disposal, reducing the need for landfills and the associated risks of air, water, and soil contamination. By converting waste tyres into valuable products, the rotary kiln reactor helps in minimizing the environmental footprint of waste disposal and contributes to a circular economy.

In conclusion, the rotary kiln reactor is an efficient and versatile technology for waste tyre pyrolysis, providing a sustainable method to recover valuable resources from discarded tyres. Its continuous operation, energy efficiency, and adaptability make it an ideal choice for industries looking to manage tyre waste while contributing to environmental sustainability. (3)

1.2 Activation of tyre char

It is a process used to enhance the physical and chemical properties of char; a carbon-rich material obtained from the pyrolysis of waste tyres. In its raw form, tyre char has a low surface area, limited porosity, and contains impurities, making it less effective for industrial applications such as adsorption, catalysis, or as a carbon material in energy storage. Activation involves treating the char to improve these properties, especially by increasing its surface area, porosity, and introducing functional groups that can enhance its performance.

There are two primary methods of activating tyre char:

I. Physical Activation

This method involves subjecting the tyre char to high temperatures (usually between 700°C to 1000°C) in the presence of an oxidizing gas such as steam or carbon dioxide (CO_2). The heat treatment opens up the pores in the char, increasing its surface area and porosity. This process helps in creating a more porous structure, which enhances its adsorption capabilities.

II. Chemical Activation

Chemical activation involves treating the tyre char with chemical agents such as acids (e.g., sulfuric acid, H_2SO_4), **bases** (e.g., potassium hydroxide, KOH), or oxidizing agents (e.g., hydrogen peroxide, H_2O_2). These chemicals help remove impurities, introduce oxygen-containing functional groups, and increase the char's reactivity and adsorption capacity. Chemical activation can be done at lower temperatures compared to physical activation and often leads to more efficient pore development. (4)

1.3 SULFURIC ACID AND HYDROGEN PEROXIDE

The use of H_2SO_4 (sulfuric acid) and H_2O_2 (hydrogen peroxide) for the activation of tyre char is crucial for enhancing its surface properties, making it more effective in applications like adsorption, catalysis, or as a precursor for carbon materials. Tyre char, a byproduct of tyre pyrolysis, contains carbon along with inorganic impurities and exhibits limited surface area and porosity in its raw form. Activation using a combination of H_2SO_4 and H_2O_2 significantly improves these properties.

Sulfuric acid (H_2SO_4) acts as a dehydrating agent and is commonly used for chemical activation. It promotes the removal of volatile impurities from tyre char and induces the formation of surface functional groups, such as carboxyl and hydroxyl groups, which increase the char's adsorptive properties. The acidic nature of H_2SO_4 also helps in removing inorganic components (like metals and ash) from the char, further purifying and enhancing its structural integrity.

Hydrogen peroxide (H_2O_2), a strong oxidizing agent, enhances the activation by oxidizing the carbonaceous material, opening up more pores and increasing the surface area. H_2O_2 introduces oxygen-containing functional groups (such as epoxides and hydroxyls), which improve the char's hydrophilicity and reactivity. Additionally, H_2O_2 can help in further removing contaminants that remain after sulfuric acid treatment, leaving behind a highly activated char with improved surface characteristics. Together, H_2SO_4 and H_2O_2 create a synergistic effect that enhances the porosity, surface area, and functional groups of tyre char, making it suitable for various industrial applications like adsorption of pollutants or as a high-performance catalyst support. This chemical activation process transforms raw tyre char into a more valuable and functional material. (5)

II. LITERATURE SURVEY

Table no 1 - Applications of tyre char

Application	Source	Reference
Tire char as adsorbent (activated carbon)	Tire pyrolysis char: Processes, properties, upgrading and applications Ningbo Gao a, *,1, Fengchao Wang a,1, Cui Quan a, Laura Santamaria b, Gartzen Lopez b,c , Paul T. Williams.	14
Tire char as supporting material and catalyst		
Tire char as reinforcing agent for tire rubber		
Tire char as battery and capacitor material		

III. MATERIALS AND METHODS

3.1 Using Rotary kiln reactor: Experimental system

The pyrolysis was carried out in a rotary kiln equipped with electrically heating coil with the inner diameter of 50 mm and length of 500 mm. This reactor was made with quartz. The reactor rotated with a constant speed of 1 rpm around its axis. In each run, about 110 g of waste tire is placed into the reactor. The proximate content of tire is: 0.8% moisture, 64.3% volatiles, 30.4% fixed carbon, and 4.5% ash. The nitrogen with ambient temperature is passed through the reactor at a flowrate of 70 cm3/min.

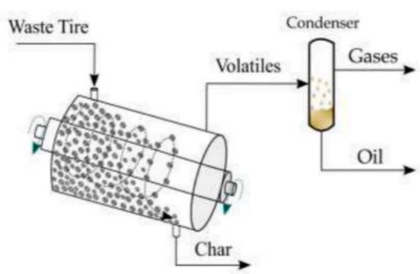

Fig 1. schematic diagram of rotary kiln reactor pyrolysis system

Source - Rotary kiln and batch pyrolysis of waste tire to produce gasoline and diesel like fuels. (3)

After ensuring that the amount of oxygen inside the reactor is negligible, the heating of the reactor is initiated. This stage is divided into two sections: (1) Temperature raising section, in which the reactor temperature will increase from the ambient temperature to the desired temperature with a constant heating rate. (2) Temperature constant section, in which the reactor temperature will maintain the desired temperature until the pyrolysis process is completed. Also, it should be noted that the pyrolysis of waste tires will occur in both sections.

Produced vapours were swept with nitrogen and after leaving the reactor, entered the condenser. The condenser temperature was maintained at a constant temperature at 25 C by circulating continuously cooling water. After condenser, products entered the separator in which liquid is separated from gas and then collected. Produced liquid was measured in each experiment by weighing of liquid in at least every 10 min. Solid was removed after the experiments ended and its weight was measured as well. (6)

3.2 Activation of carbon black

Activated carbon (AC) was prepared from waste tyre pyrolytic carbon black using one-step chemical activation method. The two different chemical activating agents, Sulfuric acid (H_2SO_4) and hydrogen peroxide (H_2O_2) were used in this study, in a 1:4 ratio.

Pyrolysis experiments were performed at various temperatures (i.e., 400–1050 C) in the nitrogen atmosphere (N_2 purity: 99.9%).

3.2.1 Activation of carbon black (CB) using sulfuric acid and hydrogen peroxide

In a typical experiment, approximately 15 g of dried-sieved carbon black was impregnated with 100 mL of H_2SO_4, and the impregnation mixture was kept for 12 hours in an oven at 100°C. Therefore, the mixture was further heated in an oven for 6 hours at temperatures ranging from 200 to 280°C. Consequently, a black bubbles-like structure of activated carbon was produced and was allowed to cool. Upon cooling, the activated carbon was rinsed with hot distilled water to remove acid from the activated carbon until the pH of the resulting solution was neutral, that is, pH = 7. The resultant activated carbon was further dried in an oven at 120°C overnight. The resultant product was thus named: Sulfuric acid-based carbon black-activated carbon (CB-AC 1). On the other hand, the hydrogen peroxide-based activated carbon was rinsed with cold distilled water to remove hydrogen peroxide residue from the activated carbon. The resultant activated carbon was further dried in an oven at 120°C overnight. The resultant product was thus named: Hydrogen peroxide-based carbon black-activated carbon. (5)

IV. RESULTS

4.1. Using Rotary kiln Reactor:

A rotary kiln reactor is one of the most efficient technologies for converting waste tires into tyre char, a valuable carbonaceous material, through pyrolysis. This continuous system provides enhanced thermal efficiency, better heat transfer, and uniform decomposition of tires, leading to high char yields with desirable properties.

The rotary kiln reactor operates at temperatures ranging between 400–700°C, ensuring optimal thermal decomposition of the rubber polymers present in tires. The rotating motion of the kiln enhances heat distribution, preventing localized overheating or underheating, which can otherwise lead to inconsistent char quality. Studies indicate that pyrolysis in rotary kilns improves carbon conversion efficiency due to better residence time control

Unlike batch reactors, rotary kilns function in a continuous mode, allowing for higher throughput and industrial scalability. This feature reduces downtime and improves process economics. The steady feed of shredded tires and the continuous removal of pyrolysis products—including tyre char, pyrolytic oil, and syngas—maximize yield efficiency.

The controlled environment in a rotary kiln reactor ensures that tyre char retains a high fixed carbon content with minimal contaminants. Proper temperature regulation prevents excessive ash formation and promotes a higher surface area, making the char suitable for applications like activated carbon production, fuel additives, and reinforcement in rubber composites.

Rotary kiln reactors allow for syngas recovery, which can be used as a heating source, making the process energy efficient. Additionally, pyrolysis minimizes landfilling of waste tires and reduces carbon emissions compared to incineration.

All runs continued for at least 120 min, in order to ensure that the pyrolysis process completely ended and no more products would be produced, the yield of produced pyrolytic oil at the end of pyrolysis process. 550 C and then yield of produced pyrolytic oil decreased by increasing temperature. This might be due to secondary reactions which could occur in higher temperatures and crack condensable produced vapors into non-condensable fractions.

The yields of gas and char at the end of pyrolysis process, mass fraction of char decreased as pyrolysis temperature increased. In other words, as temperature increased, the yield of fuel products increased as well. The important point is that the rate of increasing of produced gas was significantly accelerated at temperatures higher than 550 C. (7-10)

4.2. Characterization of carbon residues

4.2.1 Activation of carbon black (CB) using sulfuric acid and hydrogen peroxide:

The activation of carbon black (CB) using sulfuric acid (H_2SO_4) and hydrogen peroxide (H_2O_2) is an effective chemical treatment to enhance its surface area, porosity, and functional groups. This modification improves CB's performance in applications such as adsorbents, catalysts, and electrode materials.

4.2.1.1 Role of Sulfuric Acid in Activation

Sulfuric acid plays a crucial role in introducing oxygen-containing and acidic functional groups on the CB surface, such as carboxyl (-COOH) and sulfonic (-SO_3H) groups (Zhang et al., 2020). These modifications enhance hydrophilicity and adsorption properties, making CB more effective in removing pollutants like heavy metals and organic dyes. Additionally, H_2SO_4 treatment can etch the CB surface, increasing porosity and improving catalytic activity. (11)

4.2.1.2 Effect of Hydrogen Peroxide on Surface Functionalization

Hydrogen peroxide is a strong oxidizing agent that introduces hydroxyl (-OH) and carbonyl (-C=O) functional groups on CB (Wang et al., 2019). This oxidation process increases surface reactivity and improves CB's dispersibility in polar solvents, making it more suitable for composite materials and electrochemical applications. Moreover, H_2O_2 oxidation removes residual hydrocarbons, reducing impurities and enhancing the carbon purity of CB. (12)

4.2.1.3 Synergistic Effect of H_2SO_4 and H_2O_2

The combined treatment of H_2SO_4 and H_2O_2 results in a synergistic effect, significantly increasing CB's surface area, pore volume, and active functional groups. This dual modification enhances CB's adsorption capacity, making it a promising material for energy storage, environmental remediation, and catalytic applications. (13)

4.2.3 Analysis of Activation

Spectroscopic, microscopic and structural characterization methods were applied to determine the qualitative and structural properties of the carbonaceous materials in this study.

The analytical methods employed have been chosen as the most suitable ones considering the instrument, material availability, and cost. The selected methods and techniques will then be adapted to enhance the accuracy and precision of analysis of the material as far as possible.

BET analysis results revealed that activated carbon prepared via impregnation with hydrogen peroxide exhibits high surface area, high micropore volume, and average pore volume diameter. The BET results obtained confirmed the successfulness of the chemical activation method and the influence of the chemical activating agents on the surface of carbon black. (5)

V. APPLICATIONS OF TYRE CHAR

The global annual increase in the yield of waste tires, with their associated very low or even negative cost as a process feedstock, makes the consequent cost of producing tire char low, which is also the main reason for the rapid growth of the tire char market. Due to the different sources and pyrolysis processes of waste tires, tire char accounts for 30- 37 wt. % of tire pyrolysis products, with an ash content of about 7.0-15 wt. %. There are many applications that have been suggested for the use of tire char. For example, printing ink is a colloid dispersive system that is composed of filler, linking material, pigment, and auxiliary agent, where tire char could be used as pigment added to printing ink. For example, the performance of tire char as a pigment in offset printing ink. . The application of tire char as solid fuel has also been reported in the various literature. It was also suggested that tire char could be used as filler in modifying asphalt. . The specific surface area, pore diameter, and surface properties of tire char are also different when obtained under different tire pyrolysis conditions. Tire char has the characteristics of developed pores and small pore diameters. However, due to its low specific surface area, it cannot meet the application requirements of commercial carbon black. Thus, its economic value is relatively low. Therefore, attention is focused on how to effectively increase the added value of tire char. At present, the applications of tire char are wide and these utilization alternatives are represented in Fig. 2.

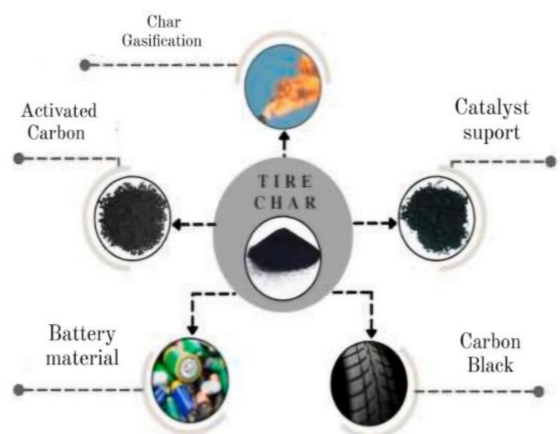

Fig 2. The main high added value applications of tire char.

Source - Tire pyrolysis char: Processes, properties, upgrading and applications

The main application includes activated carbon as adsorbents prepared by modifying tire char, rubber reinforcing materials (commercial carbon black), battery materials, catalyst support materials, and asphalt additives.

5.1 Tire char as adsorbent (activated carbon)

Carbon materials have been widely used in removing harmful substances that exist in industrial wastewater and waste gas because of their cheap and wide resource availability. Usually, activated carbon used in liquid adsorption applications should be mesoporous materials. It has been widely demonstrated that activated tire char is mesoporous, the application of chemical and physical activation processes may further develop the tire char porous structure. Tire derived carbon materials could be used as an absorbent in removing harmful substances that may exist in wastewater.

5.2 Tire char as supporting material and catalyst

The catalyst support material plays an important role in the preparation of catalysts. The properties of the support materials affect the loading capacity, the interaction between the active phase and support, and the dispersion of active components. Modified tire char has some characteristics, such as light weight, soft texture, small particle size, and large specific surface area that demonstrate its potential as a catalyst support material.

5.3 Tire char as reinforcing agent for tire rubber

Carbon black plays a reinforcing role in rubber processing, which is an important member of the reinforcing system. Carbon black added to rubber could also improve rubber wear resistance and reduce industry costs. The characteristics of tire char have a certain similarity with commercial carbon black, which provides support for its use as a rubber reinforcing additive. Modified tire char has a reinforcing effect, which enhances the mechanical properties of rubber, such as tensile strength and hardness.

5.4 Tire char as battery and capacitor material

Super-capacitors and batteries are devices for storing energy and are used in electric cars, mobile phones, computers, nano-generators, and other electronic equipment. At present, the research on activated carbon-based capacitors has attracted much attention, especially carbon nanomaterials as capacitors, have been widely studied owing to their exceptional combination of intrinsic properties. Carbon nanomaterials can be used as both matrices and functional additives due to their miscellaneous structures and dimensionality ranging from 0D to 3D. And most notably, their tunable surface chemistry and versatile surface functionalization has led to important roles in charge-transfer systems and trending energy sources. (14)

VI. CONCLUSIONS

Pyrolysis of waste tire with no metal and reinforcement fabric was conducted inside a rotary kiln reactor. The reactor was externally heated by electrical coil and rotated at a constant rotation speed of 1 rpm. The pyrolysis process was performed under nitrogen atmosphere. The reactor reached from the ambient temperature to the specified temperature and then it was fixed at this temperature until the end of pyrolysis process. It was found that the yield of the produced liquid was increased up to 550 C and then it decreased. However, by increasing the temperature, the yield of produced gas increased, while the yield of produced char decreased.

The current study presents chemical preparation, characterization and performance evaluation of pyrolytic carbon black. It was also observed that the IR spectrum of raw carbon black exhibits less functional groups as compared to the H_2SO_4-AC and H_2O_2-AC carbonaceous materials prepared. BET analysis confirmed the effectiveness of the chemical activation method and the influence of the chemical activants on the surface of carbon black. It was observed that the porous morphology of activated carbons prepared via hydrogen peroxide activation showed carbonaceous material with improved porous structure and was complimented by BET analysis results obtained.

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