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Efficient synthesis of activated carbon (AC) from biomass for catalytic systems: A green and sustainable approach
--Manuscript Draft--

From:

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Dear Prof. Bok Ryul Yoo ,

Journal of Industrial and Engineering Chemistry.

I would like to submit our manuscript entitled "**Efficient synthesis of activated carbon (AC) from biomass for catalytic systems: A green and sustainable approach** "to your Environmental Technology & Innovation for possible evaluation, consideration and publication. It is a systematic review paper which based on comprehensive evaluation of activated carbon as solid catalysts/supports to substitute homogeneous catalytic systems for the synthesis of biofuels/Chemicals. Owing to the considerable popularity of green and sustainable chemistry, renewed attention has been focused on the development of highly efficient and stable catalytic materials for producing industrially significant products from renewable resources. Therefore, applications of biomass derived activated carbon (AC) as solid catalysts/supports in various chemical transformations are promising compared to traditionally employed homogeneous catalysts with limitations of separations, disposal issues, etc. which raise environmental concerns. In view of this there is urgent call for production of these products from renewable resources which are environmentally friendly and sustainable. This review presents activated carbon (AC) synthesized from biomass as catalysts/supports. Subsequently, a number of very recent chemical transformations that show the significant potential of the activated carbon as solid catalyst/supports in renewable feedstock for the synthesis of valuable products/ biofuels were systematically reviewed. Utilization of these renewable feedstocks/waste streams will undoubtedly result to realization of sustainable synthesis and in turn usher us into circular economy through green process and design in future. This review will be of high interest to the readers of your renowned journal whose works are related to this work. Correspondence and further questions about the paper can be directed to Aderemi Timothy Adeleye, at the following address and e-mail addresses：

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We affirm that this manuscript is original, has not been published before and is not currently being considered for publication elsewhere.

Thank you very much in anticipation of your attention and consideration.

Sincerely yours,

Aderemi.

Efficient synthesis of bio-based activated carbon (AC) for catalytic systems: A green and sustainable approach

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Abstract

Tremendous efforts in developing sustainable processes for integrated production of value-added products/chemicals and fuels in biorefineries increase through delicate designs towards sustainability. This review focuses on the synthesis of activated carbon (AC) from renewable precursors and its utilisation in catalytic systems for a gentle and sustainable approach. Owing to the unique shape and porosity-controlled properties, these carbon materials could offer strong, active phase-support interactions, leading to unusual catalytic activities and selectivity in biomass upgrading. Porous carbons have been developed and used as heterogeneous solid catalysts in fine chemical and biofuels synthesis as a sustainable and economical alternative over homogeneous catalytic systems. This review revealed the AC's significance and potential as solid catalysts/supports in renewable feedstocks' valorisation. The literature showed that bio-derived activated carbon could be a promising and sustainable solid catalyst or support for producing biofuels/ value-added products with appreciable BET surface area (750 m^2/g) and total pore volume (0.37 cm^3/g). However, the surface area and pore volume vary with the treatment/nature of cellulose used as the precursor for AC production. Finally, the utilisation of these renewable feedstocks/waste streams presents us with the avenues to realise sustainable synthesis through green process and design for a sustainable future.

Keywords: Catalysis; Biofuels; Green chemistry; Sustainable Development Goals (SDGs); biorefinery; circular economy

Abbreviations

AC: Activated carbon   **VAPs:** Value added-products   **SDGs:** Sustainable development goals

UN: United Nations   **BTU:** British thermal unit   **HTCC:** Hydrothermal carbon catalyst

PKS: Palm kernel shell   **ACC:** Activated carbon catalyst   **GLY:** Glycerol

LA: Levulinic acid   **KA:** Ketone-alcohol   **CHHP:** Cyclohexyl hydroperoxide

MA: Monoacetin   **DA:** Diacetin   **TA:** Triacetin

OSAC: Olive stones activated carbon   **PSAC:** Peach stones activated carbon

SAC: Synthetic activated carbon   **PGM:** Platinum group metals

INDCs: Intended nationally determined contributions

1. Introduction

In recent years, the quest and demand for alternative, sustainable, and renewable energy sources over fossil fuels for domestic and industrial applications have been at a high rate. Interestingly, biomass has gained popularity and wide acceptance as a suitable candidate because it is renewable and durable with high utilisation potential, abundant in nature, and inexpensive. It can become a leading figure in the energy industry, one of the most sustainable energies and materials to accomplish a sustainable future [1-2]. Over the last decade, the atmospheric concentration of carbon dioxide (CO_2) has increased in the order of magnitude by more than 20 ppm, as shown in Figure. 1. [3-4]. Unfortunately, this rising CO_2 emission is associated with continuous exploitation of fossil resources such as coal and petroleum resources, which are nonrenewable hence, they get depleted with time. This and many more reasons have spurred the interest in the quest to develop new chemical technologies to transform naturally-abundant biomass to usable precursors, transport fuels, value-added chemicals, etc., to offset the effect of CO_2 emission as well as depletion of fossil fuels [5-6]. This is further strengthened by the concept of a 'zero waste economy' that targets waste biomass as a feedstock to reach this potential by transforming them into value-added materials [7-8]. A large proportion of existing oil, gas, and coal reserves must remain untapped if the average global temperature rise does not exceed 1.5 °C [9]. The origin of renewable resources has been investigated by numerous researchers and found to be remarkable as a perfect replacement for conventional nonrenewable materials. Moreover, the rapid increase in the world population has been the primary driver for promoting novel green-driven technology and processes, whose products can reduce environmental pollution and its detrimental effect on our climate. Given this, green technology research has witnessed an upsurge in research progress ranging from pilot scale to industrial commercialisation. One of the strategic pathways to excellent

biomass utilisation largely depends on the vast agro-waste, and degradable household-generated waste [10-11].
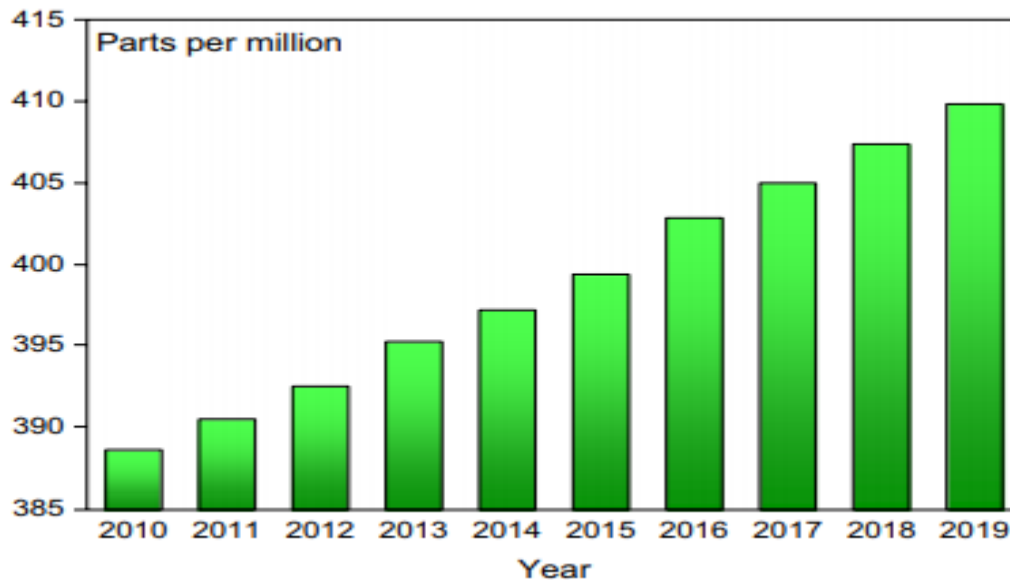
Figure 1: Average annual carbon dioxide concentration record in the atmosphere. Carbon dioxide concentration has increased by more than 20 ppm in the last 10 years. Adapted from [3], [4]

Among various biomass resources, lignocellulosic biomass represents a natural renewable and widely distributed feedstock with high efficacy to yield biofuel and value-added products (VAPs) [12]. Effective utilisation of these feedstocks can ease environmental impacts such as decreased greenhouse gas emissions, water pollution prevention, and many more [13]. Biomass waste materials are limited to lignocellulosic materials and include agricultural and forest residues, municipal solid waste (MSW), food processing wastes, and animal manures, among others [14]. Considering the potential threat to our environment by continuous utilisation of fossil resources, the potential value derived from these available sustainable wastes within the economic system cannot be ignored as an important source of bioenergy and value-added products (VAPs) [15]. Besides, important raw materials can be derived from available biomass-derived feedstocks if properly harnessed, to obtain intermediate or finish consumption [16]. Furthermore, the utilisation

of solid waste materials would ease landfill space and expedite the potential of biomass-based resources [15-16]. Biopolymers are usually composed of cellulose, hemicellulose, and lignin which exist in complex lignin - carbohydrate linkages formed due to hydrophobic and covalent interactions between lignin and carbohydrates substrate. lignocellulose is composed of 3-4 major natural polymers; phenolic polymer, cellulose (40–50 wt.%), lignin (15–30 wt.%), and also hemicellulose (25–30 wt.%) [17]. Some of the commonly used lignocelluloses in biorefinery as an alternative energy source to fossil-based fuel include woody, herbaceous plants, other crop residues like wheat straw, and sugarcane bagasse. Being the most widely utilised and distributed biomass, lignocellulose can be a renewable feedstock for biofuel production. Usually, biomass comes in different forms, ranging from waste streams, energy crops, wood, and agricultural residues, and about 170 billion metric tons of lignocellulose are produced across the globe every year. This makes them sustainable feedstocks in biofuel and chemical synthesis within economic viability and environmental sustainability [18-19]. Therefore, maximum exploration of bio-based feedstock will facilitate a positive development to attain the threshold of sustainability much needed and desired in modern habitable society [15]. Therefore, in light of the preceding, scientific investigation and processing of feedstock obtained from renewable resources have been an increasingly attractive and promising research area in the 21st century. These include but are not limited to producing degradable materials from renewable sources; a green-based novel material for various applications and green processes for chemical transformations [12,16,19-21]. The low emission of greenhouse gases is one of the advantages of energy production from biomass. A nearly zero-carbon effect to the environment is achieved [22]. Note that in biomass valorisation, the term "carbon" refers to converting CO_2 to carbohydrate and vice-versa. Some of the useful bioproducts obtained from biomass include transport fuels, platform chemicals (or their

precursors), power or heat, functional materials, etc. [15]. Relative to nonrenewable fossil resources, biomass is known for its rapid growth supply by natural environment are sustainable. Thus, it is seen as a desired resource in the recycling-based sector. The utilisation of agro-industrial waste residues has gained significant interest as renewable and sustainable feedstocks to produce fine-chemicals and related value-added products. This is driven by increasing day-by-day yearning quest for new and sustainable materials and green and zero-waste approaches [23-28]. It is worth mentioning that agricultural wastes such as vine wood [29], plantain peel and coconut shell [30], pomegranate wood [31], have been utilised as inexpensive, sustainable, and renewable precursors for the production of activated carbon (AC). AC has been identified as one of the most promising materials of the 21st century used in chemical industries thanks to its unique properties, including morphology, high mechanical strength, adsorption capacity, durability, and good chemical stability [32]. As evident from the various published works, lignocellulosic derived wastes of different sources have been reported to generate different high value-added materials used in bioenergy generation, biofuel intermediates, or liquid biofuels production purposes. Nevertheless, a literature gap still exists, requiring urgent attention as scanty reported information is available on utilising bio-derived activated carbon (AC) in greener and benign catalytic based systems for sustainable and eco-friendly production synthesis. Therefore, to adequately stimulate interest, rekindle attention, and to fulfil this literature gap, herein, intense efforts have been made to express the importance of bio-derived activated catalysts in a green and sustainable approach. With this in mind, this review, therefore, aimed to provide a comprehensive report of the most recent significant advances in the development of AC synthesized from biomass and subsequently used as efficient and eco-sustainable heterogeneous solid catalysts or supports for the transformation of biomass to value-added chemicals/products and biofuels. This is done within the

context of sustainable development and circular economy. Firstly, the emphasis was given to the synthetic methods (physical and chemical activations) for AC productions using biomass as precursors. As recently reported, the four major cases of activated carbons' catalytic applications were stressed with their catalytic performance evaluations as solid catalysts/supports. The evaluation of a catalytic system's merit was based on their catalytic performances (activity, selectivity, and recyclability). The scope and domain of applicability of AC considered are; applications of AC as a catalyst for the production of biodiesel, rich oil-based phenol, catalyst in the oxidation of cyclohexane for the production of value-added chemicals as well as supports for the catalyst was equally presented. Similarly, production of ACs, VAPs, and biofuels from biomass was also discussed as a key towards attaining a green, sustainable, and biobased circular economy.

2. Overview of Activated Carbons (AC)

Intensive research interest in carbon materials in materials science, nanotechnology, and catalysis has been inspired by their exciting properties such as texture, conductivity, stability, and hydrophobicity [32-35]. In harmony with the Paris agreement on environmental sustainability, AC could be regarded as a valuable element that will continue to gain relevance and applicability in the direct or indirect attainment of a sustainable future [36]. Interestingly, AC possesses certain morphological features such as amorphous structure [36], a large internal surface area, and a high degree of porosity [37]. This makes it a valuable carbonaceous material for diverse environmental applications. It is most abundantly found in the form of microcrystalline and non-graphitic carbon [38-39]. The principal element that makes up a carbonaceous material is carbon with the electronic configuration of carbon ($1s^2$, $2s^2$, $2p^2$). This configuration gives carbon its unique ability to form a bond with itself and other elements. It should be noted that a well-defined structure such as pentagonal, hexagonal, heptagonal, etc., can be formed as a result of the unique hybridisation (sp,

sp^2, and sp^3) of carbon. Similarly, the three most possible allotropic forms of carbon are produced: cubic diamond, hexagonal graphite, and fullerene, when carbon bonds with other carbons [40]. AC has found application in the food and beverage industries, automotive, medical, and pharmaceutical industries [41]. Most recently reported by Khaleed A. A. et al., AC with nickel oxide combined in CO gas sensing to improve conductivity and surface area [42]. Given this consideration, it is a fact that AC will remain a promising tool to facilitate the production of eco-friendly fuel and products which aimed to achieve sustainability goal and ease off climate problem-related issues [43]. If properly harnessed, the utilisation of abundant, inexpensive, and natural sources as precursors for AC production will mitigate the dependency on fossil resources [44]. AC is usually prepared by pyrolysis in an inert atmosphere to yield the so-called char, which is further subjected to a process of physical or chemical activation to increase its porosity [14]. In the past few decades, decreasing oil reserve and the increasing cost and scarcity of raw material have encouraged researchers to be searching for better alternatives from renewable resources which is cheap and benign [44]. Previous studies have revealed that lignocellulosic agro-wastes such as vine wood [29], pomegranate wood [30], soybean hull [31], plantain peel and coconut shell [45], tomato waste [30], langsat empty fruit bunch [46], papaya peel [47], and rice husk [48] are readily available, considered inexpensive, and also renewable with distinctive chemical compositions [49]. Also, activated carbon has been produced with waste tires as precursors, making this approach an effective solution for environmental pollution and economic value. It eases waste disposal issues and at the same time, serves as an economical way of making carbonaceous materials available for sustainability [50]. The various applications derived from AC utilisation have been attributed to their unique features; high surface area, a well-developed morphology, a high degree of porosity, and stable structure [32-35].

2.1. Carbon Catalysis and its Properties

The involvement of many different oxygen groups makes surface chemistry on carbon very complicated. They can be broadly classified into two major groups, namely acidic and basic oxygen groups [51-52]. The catalytic behaviour of carbon-based catalysts is often related to the oxygen-containing surface groups. The surface reactivity, mostly due to oxygen groups' presence, is an extensively studied field of carbon-based materials [52-53]. In most cases, biomass contains a significant amount of oxygen content, decreasing during the pyrolysis process under improved temperature conditions. A high concentration of oxygen in biomass could increase porosity and increased surface area after pyrolysis [54]. A contrary result has been reported based on, studies conducted on petroleum coke by Chunlan et al. This result revealed oxygen enhancement activation, which illustrates a lower surface petroleum coke with its oxygen atoms removed before activation [55]. Besides, biochar has a high carbon content (>80%) with a microporous structure. The microporous structure favours char as an effective adsorbent [56] and supports catalysts [57]. To date, there are no generally identified techniques for the production of AC from carbonaceous material with desired properties; however, CO_2 activation yields products with a narrow micropore sizes supply than when activated with steam [58-59]. Similarly, partial gasification of the carbon walls in metals' presence was found to increase the pore size as reported [59-60]. Pore size distribution and surface area are some of the most important characteristics features of catalytic applications. Likewise, product shape selectivity can as well improved by tuning the pore sizes of the catalytic materials [61]. As mentioned earlier, several biomass materials are currently being investigated as precursors in catalyst preparation and catalytic support materials. Compared with the most commonly used support catalysts such as silica, zeolites, and alumina, AC equally possesses several advantages, such as stability in both basic and acidic medium [62]. AC obtained

from biochar has the tendency to yield highly porous catalyst with surface areas ranging from 2 to 2000 m^2/g depending on the source and biomass activation method [63] in comparison with other common catalytic support materials surface areas for example, γ-alumina {150 to 400 (m^2/g)} [64], silica {300 to 750 (m^2/g)} [65], zeolites {460 to 800 (m^2/g)} [62], and carbon black {25 to 1500 (m^2/g)} [66]. By altering conditions activation, the pore size arrangement of the AC can easily be influenced by modifications. Thus, this tunability is essential since the pore size distribution controls the reactant's flow, favouring the reactant and isolation of the desired product. The surface functionality, on the other hand, impacts the AC performance [67]. These features allow for easy conversion and fabrication of materials with different shapes and dimensions. In general, carbon materials have been used as supports for PGM (Platinum Group Metals) catalysts while at the industrial level it is also being utilised as PGM support, most importantly in hydrogenation reactions [62].

3. Synthesis methods for the production of activated carbon (AC)

 Preparation and production of AC can be carried out through direct activation of dry raw precursor or two-stage techniques involving initial carbonization process followed by activation. The first step requires carbonization of the raw materials at high temperatures. During this process, the materials are exposed to a red spot (less than 700 °C) temperature, which leads to evaporation and discharge of the hydrocarbon's species via the distillation apparatus in the absence of oxygen. In a nutshell, the carbonization process is pyrolytic, which resulted to the production of carbonised material in the form of either char or biochar [68-70]. At the end of AC activation, several other activation methods could be subsequently employed to influence porosity and create structures for the formation of fine solid cavities in activated carbon [71]. According to the International Union of Pure and Applied Chemistry (IUPAC), these pores on the surface of support material can be

classified into micropores (< 2 nm), mesopores (2 nm - 50 nm), and macropores (> 50 nm) [69]. AC could be prepared in two ways: physical and chemical (details are discussed in the subsequent sections).

3.1 Production of activated carbon by physical activation

As stated earlier, physical activation involves a two-step process. Firstly, a carbonization protocol of dried biomass samples between 400-700 °C for the production of biochar and then accompanied by activation step where oxidising gas such as steam, air, CO2 or their mixtures are used [71] at a range of temperature (800-1100°C) to carry out burn off. However, the obtained biochar through carbonisation does exhibit a low surface area of less than 300m2/g [72]. The low surface area and adsorption characteristic features are connected to pore blockage due to generated tar. The removal of this tar can be removed application of activation. Both carbonisation and activation can be simultaneous while the temperature is kept in the range of 600-800 °C [73] [74]. Compared with the dual-stage process, the single-stage process involves the dried sample to be subjected to carbonization only with an optimum temperature being attained. The biochar is subjected to further thermal treatment to the desired duration by switching from inert to oxidising gas [72]. Hence this method results in the usual widening of the narrow pores produced on the surface of biochar during physical activation, and as well new pores are generated, which positively influences the porosity and surface area of the carbonaceous porous structure [75]. Physical activation tends to produce AC with better quality, albeit with relatively lower carbon yield than chemical activation due to higher activation temperature and time during the whole process [76]. In physical activation, steam and CO_2 are commonly the preferential activating agents used because air (oxygen) usually incur exothermic reactions which can cause excessive burnings, both on the internal and external surfaces of the biochar and this subsequently results to the formation of a porous structure

according to the Equations 1–3 [72-75,77]. Rafsanjani and co-workers hypothesised that water molecules could diffuse into pores of biochar more quickly because of the smaller size of H_2O than CO_2; [76] and consequently leads to a more rapid reaction than that of carbon with CO_2, this directly leads to the development of new pores [78]. AC exhibits a larger surface area in a limited time when steam is employed for the activation process [79]. According to the published reports by Chowdhury and his co-workers, the four mechanistic carbonisation stages are summarized in table 1 [77].

$$C + O_2 \longrightarrow CO_2 \quad \dots\dots\dots\dots\dots\dots\dots\dots\dots\dots(1)$$

$$C + CO_2 \longrightarrow 2CO \quad \dots\dots\dots\dots\dots\dots\dots\dots\dots(2)$$

$$C + 2H_2O \longrightarrow CO_2 + 2H_2 \quad \dots\dots\dots\dots\dots\dots\dots\dots(3)$$
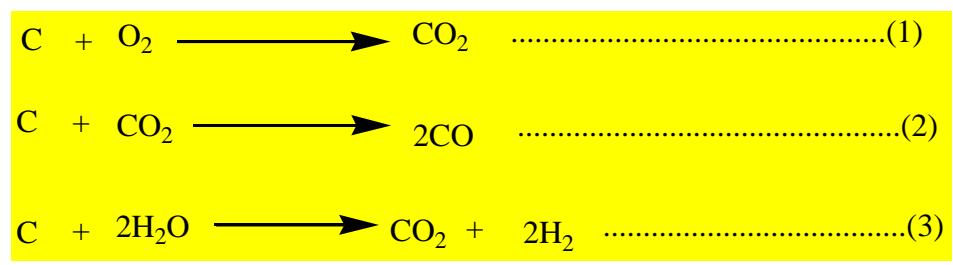
Table 1: Four stages of the carbonisation process

Carbonisation Stage	Reaction Temperature(K)	Nature of reaction	Process
Stage I	≤ 473	Endothermic	This stage involves drying of precursors (i.e raw materials) in order to remove the existing moisture
Stage II	443-573	Endothermic	This stage is known as pre-carbonisation which involves production of some pyroligneous liquids (such as acetic acid and methanol) and light tars and non-condensable gases (CO and CO_2)

| Stage III | 523-573 | Exothermic | The removal of pyroligneous liquids and light tars occur in this stage to enhance the production of biochar |
| Stage IV | >573 | - | The 4th stage involves the elimination of volatile and non-carbon species to enhance the fixed carbon content of biochar |

AC exhibits a larger surface area in a limited time when steam is employed for the activation process [80]. Comparatively, CO_2 activation also influences the creation of new pores in the materials. In contrast, steam activation favours micro-pore widening towards the formation of mesopores and macropores from the initial stage of the activation process at a selected heating rate [75]. When a high activation rate is adopted in the reaction process, it will produce a higher percentage of mesoporous and microporous structures for both steam and CO_2 activations, respectively [72]. In another reports, Rodríguez-Reinoso et al. and Zamora and co-workers studied the effect of the different physical activation gases on the development of porosity from olive stone-derived chars. Both CO_2 and steam were employed in their study. The activating agents CO_2 and steam produce different changes in the porous structure of the char when olive stone was used as the precursor in the synthesis. The results obtained show that CO_2 activation causes mainly the creation of micropores. With water steam, although for a given burn-off the porosity development reached is higher than that corresponding to CO_2, the carbons present remarkable wider pore distributions (likewise, the obtained volumes of macropores when steam activation was used are significantly higher). In all the cases an increase in activation reaction time resulted to a widening

of the pores [81,82]. Hence using CO_2 activation is desirous owing to its cleanness, handling gas flow does not come with difficulty. In contrast, the activation activity can be monitored at about 800 °C at high temperatures due to its slow reaction rate. Activation by carbon dioxide creates a larger number of groups evolving as CO and these groups are thermally more stable than those produced by steam activation [82, 83,84-86].

3.2 Activated carbon production through chemical activation

Chemical activation is often performed using cellulose-based precursors such as wood, sawdust, fruit pits, etc. During this process, e.g., (preparation of activated carbon), the precursors which are usually organic raw materials are activated in the presence of chemical species at elevated temperatures [70,87]. Before the process fully begins, the raw materials are initially saturated with an oxidising agent and further dehydrated chemicals. After impregnation, the suspension gets dried with the remaining mixture followed by thermal treatment at a given/selected optimum reaction time. Cellulose is degraded at this stage because activation occurs at temperatures in 400-900 °C which is considered suitable enough to perform degradation on cellulose. This is often determined by activating material and the properties of the final product. Afterwards, the active material which in this case is activated carbon is recovered after washing and rinsing the resulting mixture severally [87]. Also, chemical activation agents are dehydrating agents that activate pyrolytic degradation, improve the activated carbon yield, while causing changes in the thermal precursor's decomposition, giving rise to the formation of porous carbon materials [88-89]. Gratuito et al., (2008), in their work, opined that the increase in surface area is brought about by the unique ability of the activating agents to penetrate deeply into carbon structures, thus initiating the formation of small pores in the activated carbon. Unlike thermal physical activation, both carbonisation and activation occur concurrently during chemical activation. Elsewhere, physical

activation employs two different furnaces, while chemical activation occurs in a single furnace [87]. Some of the factors that influence the properties of the final AC during chemical activation include; the ratio of chemical agents to dry precursor and impregnation [89]. Interestingly, activated materials interact chemically with carbon matrices to obtain gas products that yield a porous structure [89-90]. However, several washing steps required for the activating agent from the synthesized product are considered significant drawbacks. In the washing stage, toxic wastewaters are generated, which has been seen as a potential threat to the environment if not adequately treated [91-94]. Good selection of synthetic parameters for quality activated AC production is another factor that needs to be considered for the success of chemical activation activities. During this stage (production of AC), the process's efficiency and reliability are considered an important index [89, 93-94]. As mentioned earlier, the merit of chemical activation methods is attributed to lower reaction temperatures and the shorter time required to complete the carbon materials' activation process. When activated carbons are used as catalysts or catalyst supports for reactants with large sizes, diffusion constraints and accessibility to active sites become an obstacle. To overcome the constraints mentioned above, an enormous effort has been made to develop synthetic methods for useful mesoporous carbons [91-94]. Given these considerations, different chemicals (see Table 2) have been investigated with each reacting differently with precursors, thus, affecting the general characteristic adsorption behaviour [36,95]. The two methods have their advantages. For example, physical activation has an advantage over chemical activation because it avoids incorporating additives/impurities coming from the activating agents. Microporous, as well as ultra-microporous carbons, are usually formed by this method. On the other hand, chemical activation is preferred over physical activation due to the lower temperature and shorter time needed to activate materials. The carbons formed by this method generally have

wider micropores and higher pore volumes, making them comparatively more suitable for liquid-phase adsorption. [92].

Table 2: Potential chemical activators

Activator Groups	Examples
Alkaline	potassium hydroxide (KOH), sodium hydroxide (NaOH), calcium chloride (CaCl$_2$), and potassium carbonate (K$_2$CO$_3$)
Acidic class	phosphoric acid (H$_3$PO$_4$) and sulphuric acid (H$_2$SO$_4$)
Intermediate metal salts	Zinc chloride ZnCl$_2$, Ferric chloride (FeCl$_3$)

The advantages and drawbacks of the carbon chemical activation are presented in figures 2 & 3 respectively [86].
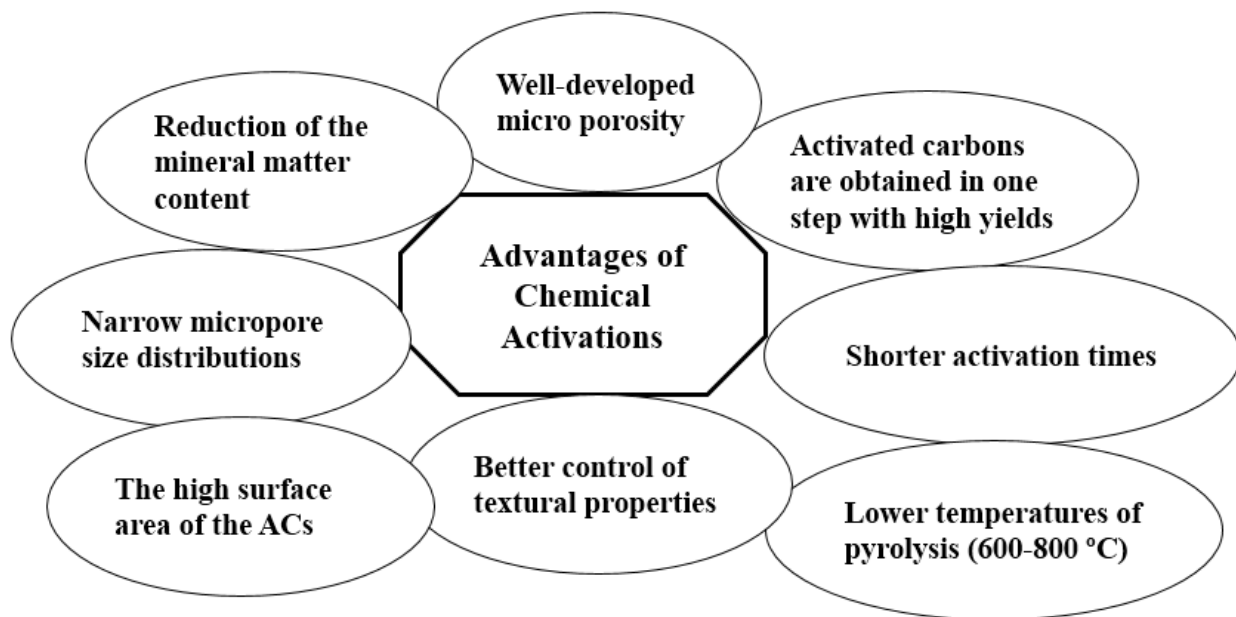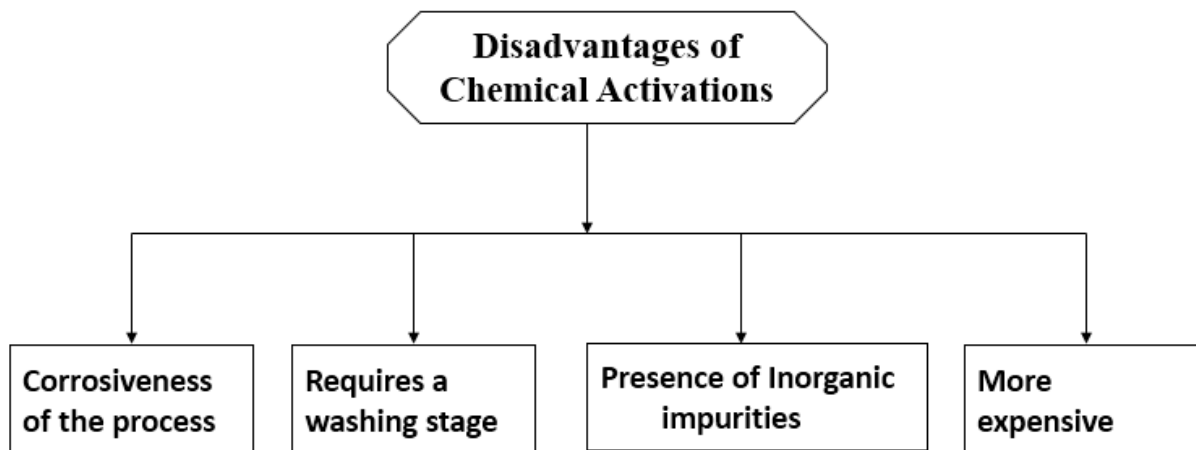
Figure 2: Chemical method Advantages

Figure 3: Disadvantages of Chemical method.

4.0 Characteristic properties and characterization techniques for the novel activated carbon

The commonly used characterization techniques for the novel activated carbon and desired characteristic properties that determine their suitability and performance are summarized in table 3

Table 3: Characteristic properties and characterization techniques for the novel activated carbon

Commonly used characterization techniques for AC	Brief Notes	Ref.
Determination of pore size and surface area of AC via S_{BET}.	The useful information about porous properties of the prepared activated carbon can be determined by a nitrogen adsorption method	[96]
Elemental composition by CHNS/O:	In order to be able to give adequate comparison to the various operating parameters (such as the influence of the different preparation, activation and modification methods on the carbon or heteroatom content) the elemental composition for both precursors (i.e starting materials) and AC produced needs to be determined.	[96]
FT–IR Spectra of Activated Carbon	The surface chemistry of an AC can be determined by the type, quality, and as well through the existing bonding of functional group available. FT-IR	[92]

	spectroscopy is suitable to study the useful information regarding the identification of functional group and also provide available information about the chemical structure of the synthesized AC. Therefore, adsorption capacity of activated carbon depends upon porosity as well as the chemical reactivity of functional groups at the surface. Knowing the functional groups would really help to estimate the adsorption capability of the novel activated carbon obtained.	
X-Ray Diffraction Analysis (XRD)	XRD is the useful characterization technique suitable to obtain information on the nanoparticle size and crystalline and amorphous materials. From the published works on X-Ray Diffraction analysis of activated carbons, predominantly amorphous solid with large internal surface area and pore volume have been reported. Likewise, the synthesized activated carbon exhibited disordered microcrystalline structure in which graphitic microcrystals are randomly oriented.	[97]

Textural characterization by scanning electron microscopy (SEM) analysis	SEM is a useful characterization technique in surface chemistry. SEM has really helped in understanding the surface morphology of starting materials and the activated carbon synthesized. The useful information is provided using a SEM for the identification of the porous structure and external surface of the AC. The development of the pore system in the synthesized activated carbon (AC) is mainly dependent of the precursor employed during production process. Likewise, the average amount of pores is mainly determined by the concentration of impregnation agent used.	[98]

5.0 Green Chemistry: Catalysis, Renewable feedstocks, and Sustainability

As reported by Anastas and Warner in their well-known report called "12 principles of Green Chemistry", Green chemistry is considered the key driving force for pollution prevention [99]. To attain sustainability creation of a feasible environmentally-friendly chemical process, the development of novel and cost-effective methods capable of mitigating environmental degradation is obligatory and inevitable. As the name implies, green chemistry aimed to minimise or limit the build-up of noxious substances in designing, manufacturing, and applying chemical products. The difficulties encountered in attaining sustainability hassled to new technologies that offer the society with eco–friendly products [99-102]. Undoubtedly, advances in green chemistry will help identify those menace and minimise their detrimental impact on the environment [99-102]. A

catalyst lowers the energy cost of converting molecules and alter the reaction pathways toward the final product distribution stage. Advances through modification in catalyst technologies have played a vital role in searching for a sustainable modern society and an eco-friendly planet. It is forecasted that global energy consumption will rise by nearly 50% to over 900 quadrillion British thermal unit (BTU) between 2018 and 2050. A large portion of the worldwide energy consumption will go into the production of chemicals fuels. Because of this, progress in catalyst development can minimise competing demand for both energy consumption and waste side products. Thus, energy and environmental challenges require the design of environmentally benign catalysts [103-104]. Heterogeneous catalysts account for a larger percentage of catalyst used at an industrial level within the catalytic community and constant catalyst evolution and mutation. Catalysts have been found useful in various chemical processes and represent the pivotal optimisation step towards achieving sustainability, both economically and environmentally. In particular, solid catalysts have been considered an essential part of the green chemistry approach for sustainable advancement [105]. Therefore, strict government regulations have forced chemical industries to explore alternative and sustainable catalytic materials. Thus, heterogeneous catalysis, where the catalyst exists in a different phase (typically solids) as the reactants (mostly liquids or gasses), could offer exceptional and tremendous potentials for many energy and environmental-related applications including biomass upgrading [103-104]. Some of the advantages of heterogeneous solid catalysts are represented in figure 4. It is worth mentioning that solid catalysts are recyclable; hence, they can be recovered efficiently from reaction mixtures and reuse in multiple catalytic cycles, making the process cost-effective and sustainable unlike homogeneous catalysts [104].
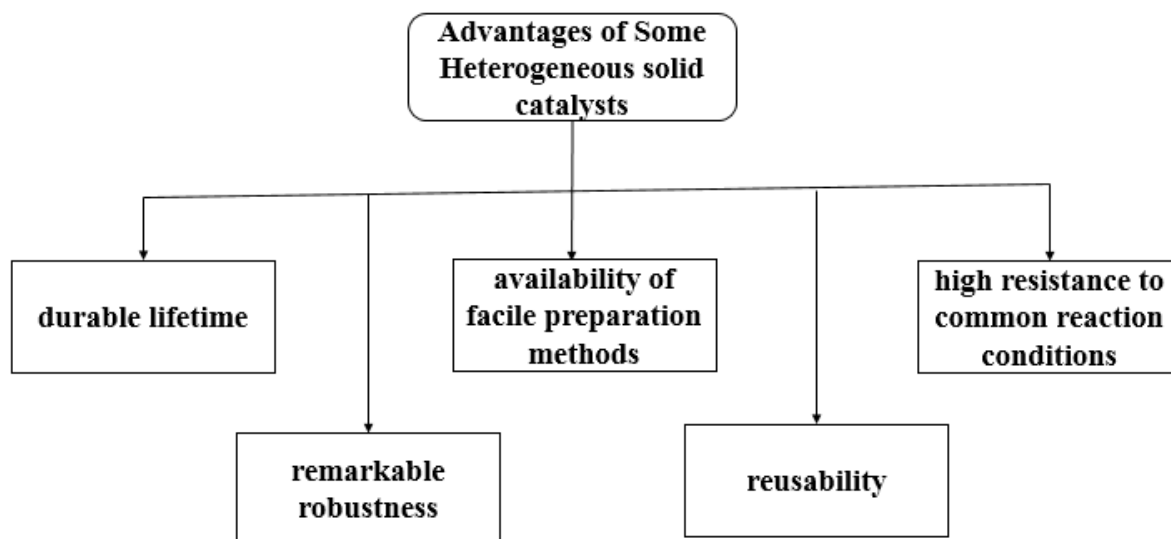
Figure 4: Some of the advantages of heterogeneous solid catalysts.

In light of the several drawbacks encountered by the traditionally employed catalysts in several procedures, much research effort has been given to the development of novel and improved heterogeneous catalysts in recent years. On the other hand, setbacks such as high reaction temperature, prolonged reaction time, low conversion, low regioselectivity, lack of usage, and difficulty in separation and purification of the products hamper the development of homogeneous catalysts [105]. As the campaign to promote sustainable and eco-friendly chemical approaches increases, renewed attention has focused on the fabrication of unparallel, efficient, and stable catalytic materials to produce Eco-friendly products from renewable energy feedstock [106-107]. Given the environmental impact of the chemical processes in recent years, the catalytic reaction is considered the key driving force of chemical industries for the future. Moreover, over 90% of these processes employ the use of catalysts worldwide to facilitate productivity.

Interestingly, new and exciting opportunities are emerging in this field day by day to develop cost-effective, energy-efficient, highly active, selective, and environmentally benign heterogenized

systems for sustainable industrial products [103,108]. Currently, carbon materials are considered materials of high value in industrial catalytic processes, and their potential has not been fully exploited. Although, in recent decades, we have witnessed unprecedented interest in carbon nanomaterials, however, the reported applications have shown that more is needed to be explored within catalytic space as scarce information is available on its exploitation. Some fundamental issues associated with the surface area, surface chemistry, and structure must be addressed to the fullest for large-scale applications. Generally, carbon supports are attractive for heterogeneous catalysis because the carbon surface is more inert than in typical oxide supports. Similarly, carbon materials offer an extensive range of applications and unparalleled flexibility in tailoring their physical-chemical properties for specific needs. Although, several factors that improve catalyst activity are associated with both the physical properties of carbon (high surface area, porous structure) and the chemical properties (oxygenated groups, sulfonic acid groups) [109].

6.0 Catalytic applications of Activated Carbon (AC) in Biorefinery

The technology for the production of chemicals has not changed drastically over the past decades. The fact remains that ancient stoichiometric technologies are still widely employed for the production of chemicals (especially high value-added chemicals) such as acid/base-catalysed reactions with mineral acids (H_2SO_4, H_3PO_4, etc.), Lewis acids ($AlCl_3$, $ZnCl_2$, etc.), and inorganic bases (NaOH, KOH, etc.). However, stoichiometric reagent's major drawback is the difficulty associated with recovery and recyclability of the used chemicals after a complete reaction. Therefore, replacing these stoichiometric methodologies with a green-based catalytic procedure is imperative [100,106, 110-111]. To this end, many soluble homogeneous catalysts have been designed and synthesized for the green chemicals processes. These catalysts are usually dissolved in reaction media, making all catalytic sites accessible to substrates, resulting in high catalytic

activity and selectivity [107]. However, there are several environmental challenges associated with partially spent liquid acid disposal into the environment, resulting in an enormous waste of time and money to remediate the affected areas [112]. Given these considerations, manufacturers are constraint to look for sustainable and environmentally benign feedstocks as an alternative following environmental compliance as proposed by the environmental protection agency [112]. It should be noted that proper understanding of the pore structure's chemistry is required for the design of catalysts for such biomass transformations to minimise mass transport limitations, improve hydrothermal stability under aqueous operation, and consequently, tunable hydrophobicity to aid product/reactant adsorption [113]. On the other hand, carbon materials exhibit several merits when used as supports in heterogeneous catalysis [109]. It is worth mentioning that the development of easily recoverable and recyclable solid catalysts has attracted attention in this application domain, particularly in eco-friendly syntheses of high value-added chemicals, biofuel intermediates, and biofuels. To this end, AC is one of the components of carbon materials suitable for functioning adequately concerning the benefits above and attractive properties as listed below [109].

Advantages of carbon materials as supports in heterogeneous catalysis

- ❖ The carbon surface is relatively inert, preventing unwanted reactions catalysed by the support surface or reaction of the support with the active phase.

- ❖ Tailored pore size distribution for specific reactions.

- ❖ Although carbon is usually a material with a hydrophobic nature, their surface's chemical nature can be modified chemically to give them some hydrophilicity.

- ❖ The active phase, usually expensive, can be easily recovered by simple calcination of the support.

The physical properties of Activated Carbon (AC) are summarized in Table 4 [109].

Table 4: Physical properties of Activated Carbon (AC)

Physical properties of Activated Carbon (AC)
A. **Specific surface area (m² g⁻¹):** 1000−3500
B. **Pore volume (cm³ g⁻¹):** 0.6−2
C. **Density (g cm⁻³):** 0.4−0.7
D. **Electrical conductivity (S cm⁻¹):** 0.3−10
E. **Cost:** Low

6.1 Activated Carbons for the synthesis of biodiesel

Undoubtedly, solid base catalysts are easily designed and synthesized, corrosive free, cheaper, and follow a more environment-benign operations pathway [16, 114-115]. Besides, a less to moderate amount of solid-based catalyst is required to perform transesterification activities. This is in sharp contrast with homogeneous processes, for example, in the production of 8,000 tonnes of biodiesel, 88 tonnes of sodium hydroxide may be required as reported [116]. Similarly, 5.7 tonnes of solid-supported MgO were needed to produce 100,000 tonnes of biodiesel, according to [108]. Metal oxides, mixed metal oxides, zeolites, hydrotalcite, etc. have also been used as heterogeneous catalysts for biodiesel production [118]. However, the formation of three phases together with oil and alcohol hinders the usage of these metal oxides; thus, limits diffusion by lowering the rate of the reaction [116]. However, researchers have used co-solvents, such as n-hexane or tetrahydrofuran (THF), to overcome this mass transport limitation. These co-solvents enhance the mixing of oil and methanol into one phase, thereby increasing the reaction rate. Another way to

overcome mass transfer in the solid catalyst is to use a support system that offers more specific areas and pores for active species, anchoring and reacting with large triglyceride molecules [119]. Recall the significance of materials with large surface area and recently a report published by Konwar et.al, shows that waste shells of T-striatula were the basic raw material used for the production of activated carbon and CaO. The AC-supported CaO catalyst was used to transesterification of waste cooking oil with a better yield of over 96% of methyl ester conversion under an adopted optimum condition. Furthermore, the catalyst's reusability was also investigated and recovered successfully, which might not be possible with homogeneous based catalysts. It was found that the catalyst was reusable and retained its initial catalytic activity for about five entry cycles. Hence, it can be inferred that the catalyst, besides unusual catalytic activity, was also reusable, and this is advantageous in real or industrial applications [120]. As already mentioned, AC is a material of multi-purpose values. In another published report, AC was prepared using spent tea waste as a precursor and subsequently applied for the purification of crude biodiesel manufactured from waste cooking oil (w.c.o). To verify the viability of AC, silica gel, and water washing were also applied biodiesel purification. However, the study showed that in general, using AC for biodiesel purification gave higher yields and better fuel properties compared to the biodiesel washed using silica get and water. Moreover, the reported fuel properties were in per the ASTM standards. To further explore its characteristic regeneration feature, spent AC was subjected to regeneration activity and further reused. The result showed that regenerated AC was outstanding in synthesising biodiesel fuel with high yield and appropriate required fuel properties compared to the other reported methods [121]. In another study, Memon and his co-workers prepared the low-cost hydrothermal carbon catalysts (HTCC) from Ziziphus Mauritania L. biomass. Subsequently, they applied it as a solid catalyst for biodiesel production [122]. Ziziphus

Mauritiana L. is widely distributed in the tropical and subtropical regions of the world. It belongs to the Rhamnaceae family [123]. Its fruit's pericarp is consumed either fresh or dried, while its seeds are usually discarded. Leveraging on the vast discarded seeds and examining the oleic acid content with methanol via batch to batch study over HTCC was studied. The surface area and acid density of prepared HTCC were found to be 15.97 m2 g−1 and 2.53 mmol g−1, respectively. Statistical analysis was performed by using ANOVA, mean, standard deviation, and variance methods. The maximum oleic acid conversions from methanol were obtained up to 91%. Furthermore, reusability and regeneration studies were also carried out to assess catalytic performance. The study indicated that HTCC obtained from Ziziphus Mauritiana L. is an excellent catalyst and can be used as an alternative for the esterification of oleic acid to produce alternative-diesel [124].

6.2 Activated Carbons for the synthesis of phenol-rich bio-oil

Phenol is considered an important precursor in various useful chemical products, such as phenolic resins, alkylphenols, etc. However, the current technology to produce phenol comes at a huge cost of fossil energy consumption and environmental contamination, mainly through the industrial conversion by the cumene process from benzene [124-125]. Fortunately, recent advances in pyrolysis have revealed that phenols can be obtained from the bio-oil derived from the pyrolysis of lignocellulosic feedstocks. Undoubtedly, this offers a new route to manufacture phenols from renewable bioresources [126]. The phenols are thought to be mainly generated from the decomposition of lignin. However, producing phenol-rich bio-oil from the conversion of lignocellulosic biomass has its setback. The low content of phenols in bio-oil causes the down-stream expensive extraction and purification process combined [127]. Several efforts have been put into research to enhance phenols' production by using catalysts during the pyrolysis process.

To this end, activated carbon catalyst (ACC) has been recognised as an effective catalyst in selectively converting lignocellulosic biomass into phenols [127-128]. In their study, AC obtained from corn Stover by microwave/H_3PO4 activation was effective in the catalysis of Douglas fir sawdust to synthesise simple phenolic compounds. Taguchi method suggested the optimal condition for AC preparation was phosphoric acid to biomass ratio of 0.8, microwave power of 600 W, and the microwave time of 20 min. The incorporation of corn stover derived AC significantly increased the yield (2.46 mg/ml) and selectivity (up to 75%) of phenols. Moreover, ACs prepared from higher phosphoric acid impregnation ratios resulted in higher contents of phenolic compounds. The FTIR, SEM/EDX, and NH3-TPD results revealed the presence of phosphorous-containing acidic surface functional groups, which provide active sites for the selective production of phenolic compounds. The higher acid impregnation ratios and higher microwave irradiation power led to the widening of the microporous structures during the activation process [129]. Accordingly, the previous studies by Bu, Q et al., ACC demonstrates good performance in phenolic-rich bio-oil production during the lignocellulosic biomass pyrolysis process [130-131]. High-purity production of phenol can be achieved when H_3PO4-AC is used as a catalyst during the glucose pyrolysis process. However, the absolute content of phenols in bio-oil was still not high, only around 50 mg/mL. Therefore, the practical fulfilment of lignocellulosic biomass claims effective technologies to achieve high-value-added chemicals' mass production. Furthermore, the conversion of lignocellulosic biomass into transportation biofuels, such as jet engine fuels, gasoline, and diesel has also received great attention worldwide, as reported [132-133]. The catalytic co-pyrolysis of Douglas fir and low-density polyethylene with commercial AC catalysts was studied for the first time. Therefore, six types of ACC were investigated and compared. The obtained liquid product contained physically distinct parts in the oil and water

phases, where the percentage of the oil phase ranged from 10.10 to 64.4 wt.% depending on various co-pyrolysis conditions. The oil phase of bio-oil was rich in C8-C16 aromatics and aliphatic compounds (up to 98.6 area. %) that were well suited for transportation jet fuel. The bio-oils main constituents in the water phase comprised phenols and guaiacols. The high phenols selectivity (up to 92.9 area. %) and phenol concentration (up to 26.4 mg/mL) were obtained. The main fractions of gaseous products obtained during this process were hydrogen, methane, carbon dioxide, and carbon monoxide, where a high concentration of methane (23.6 vol.%) and carbon monoxide (39.1 vol.%) could be obtained. The process was optimised based on bio-oil yield, C8-C16 hydrocarbons selectivity, and phenol concentration. The work was considered novel, eco-friendly, and sets the pace for a cost-effective route in the synthesis of phase-divided phenols-enriched chemicals and transportation of jet fuels from biomass over ACC [134]. Another research team also addressed the in-situ microwave catalytic reforming of volatile matter from palm kernel shell (PKS) over iron-loaded activated carbon (Fe/AC) catalysts. The effects of catalyst composition on the secondary gas-phase reactions and products' distribution were investigated at 500 °C. It was observed that the Fe/AC catalyst facilitates the yield of light gases. Using the 1%-Fe/AC catalyst, the yield of gaseous fraction peaked at 37.09 wt.%. The selectivity of the deoxygenated products was promoted in the presence of Fe. Catalytic reforming of PKS pyrolysis vapours over Fe/AC strongly improved the generation of phenol and H2; the concentrations reached 75.09 area% and 75.12 vol.%, respectively. Catalytic pyrolysis of syringol and guaiacol as model compounds showed that Fe/AC catalyst enhanced the demethoxylation and deoxygenation reactions to generate phenol via active Fe sites [135] selectively. Similarly, AC was also effective for producing 4-ethyl phenol from bagasse with high selectivity (28.8%) [136]. In another similar work, Huo and his co-workers used biomass-derived activated carbon (BAC) and MgO as catalysts

for the catalytic pyrolysis of dumped waste plastics to produce renewable and sustainable jet fuel and hydrogen. The published results revealed that BAC and MgO as catalysts had an outstanding performance in catalytic conversion of waste plastics to produce renewable hydrogen and jet fuel. 100 area. % jet fuel range products were obtained under-investigated experimental parameters [137]. It can be concluded from their reports that an effective, convenient, sustainable, and economical route was made available for the synthesis of renewable jet fuel and hydrogen from waste plastics over BAC (equally sustainable, renewable, and eco-friendly catalyst) and MgO catalysts.

6.3 **Application of Activated Carbons in the oxidation of cyclohexane to value-added chemicals (VACs)**

In a recent development reported by Sadiq M et.al, AC was synthesized from peanut shells and was employed as a metal-free catalyst for the solvent-free oxidation of cyclohexane to cyclohexanol and cyclohexanone. The mixture is known as KA (ketone-alcohol) oil which connotes ketone-alcohol and is usually performed in oxygen under moderate reaction conditions. The heterogenized systems display good activity and high selectivity in the production of KA (ketone-alcohol) oil (cyclohexanol and cyclohexanone mixture) from microwave-assisted oxidation of cyclohexane, and in turn allowed their easy recovery and recyclability, at least for four entry cycles. The reaction parameters were improved, and the catalytic efficiency was examined based on maximum productivity (2.14 mmolg^{-1}h^{-1}, without base, and 4.85 mmolg^{-1}h^{-1}, with 0.2 mmol NaOH). Accordingly, AC shows recyclability for several cycles without any vital loss of catalytic activity. The ease of preparation, separation, exciting activity, low cost, non-

leaching, and eco-friendly nature makes AC an indispensable catalyst for solvent-free oxidation of cyclohexane in their study [138]. Oxidation reactions are essential in the chemical industry to produce many essential new compounds. The development of homogeneous catalysts for cyclohexane's selective oxidation, a high significance reaction in the chemical industry, has been extensively studied in recent times [139]. The oxidation of cyclohexane to cyclohexanol and cyclohexanone (KA oil) is of utmost importance due to their applications as precursors in the synthesis of Nylon-6,6 and Nylon-6, solvent lacquers, varnishes, and shellacs, and as homogeniser and stabilizer in soaps and detergents [140-141]. At an industrial-scale, the process of KA oil production is performed in two consecutive-step: The noncatalytic autoxidation of cyclohexane to cyclohexyl hydroperoxide (CHHP) in the presence of oxygen, followed by the conversion of CHHP to KA oil using a homogeneous cobalt (Co (II)) catalyst. However, this process is less effective as a result of low cyclohexane conversion of $3-4\%$ with the selectivity of $60-70\%$ to CHHP [142], the huge operation cost of recycling of unreacted cyclohexane, partial recovery of Co(II) catalyst due to its easy discharge with the salts leaving the process, and use of 180 Kg of NaOH to produce a single Kg of KA oil are some of the drawbacks of this process which collectively generates about 1100 Kg of alkaline waste [143]. The constraints were overcome as reported in the work of Sadiq M and co-workers. The application of AC minimized the environmental and economical drawbacks of the industrial scale selective oxidation of cyclohexane to KA oil as a heterogeneous solid, environmentally benign catalyst. The reaction pathway is shown in scheme 1 [138]. In another study, C-scorpionate iron (II) complex [FeCl2(Tpm)] [Tpm $= \kappa 3 -HC(C3H3N2)3$] was immobilized on three nanoporous carbon supports to produce active, selective, and recyclable catalysts for the oxidation of cyclohexane. The effect of the porous carbon supports on the performance of the heterogenized [FeCl2(Tpm)] catalyst was

studied using materials with different porosity: microporous (GL50-ox, wet oxidized GL50 Noritz sample, and S, sisal-derived AC prepared by chemical activation), and mesoporous (CMK-3) materials. The heterogenized systems exhibited good activity and relatively high selectivity to forming KA oil (cyclohexanol and cyclohexanone mixture) from microwave-assisted oxidation of cyclohexane. They allowed their easy recovery and recyclability, at least for four run cycles.
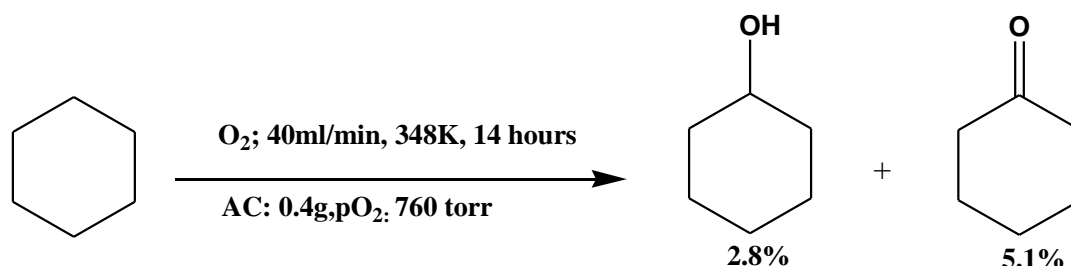
Scheme 1: Transformation of cyclohexane to KA oil over AC catalyst (2.8% cyclohexanol and 5.1% cyclohexanone were obtained from oxidation of cyclohexane over renewable AC)

The stability of the hybrid materials and pristine carbon materials were investigated based on their recyclability in consecutive catalytic cycles. After the 2nd cycle, all catalysts retain ca. 80% of their initial activity. However, after the 3rd successive cycle, the activity drops to ca. half of the initial value. After the 4th cycle, the heterogenized materials still present 35 and 20% of their initial activity, for Fe@GL50- ox and Fe@CMK-3, respectively. Carbon CMK-3 offers at the end of the fourth cycle was around 20% of its initial catalytic activity. For the heterogenized materials, the decline of catalytic activity is mostly attributed to iron leaching to the solution. And in the case of the pristine carbons, the activity decay is due to the blockage of the active sites on the surface of the materials, limiting the accessibility of the species to the inner pore network. This work's most important result was discovering significant self-catalytic activity observed for the pristine carbon

materials GL50-ox and CMK-3 towards cyclohexane oxidation under Microwave irradiation conditions-in the absence of the iron complex [139].

6.4 Applications of Activated carbon as support

Apart from acting as a catalytic material, activated carbon was also employed to support metals, organocatalysts, or organometallic catalysts in several reactions [144-145]. Efforts are currently ongoing, aimed at the heterogenisation of homogeneous active species to combine the benefits of homogenous catalysts with well-known active centres with the Hydrogenation reactions in the presence of Pd catalysts [146-147]. Prati et al., in their study, synthesized AC from three different wood species, namely pine, birch, and spruce sawdust. After carbonization and activation, the samples exhibited a high surface area (>1000 m2 g -1) and comparable mesoporosity. Afterwards, the synthesized carbons were then employed as support materials for AuPt nanoparticles and investigated for glycerol (GLY) oxidation in the liquid phase and the hydrogenation of levulinic acid (LA) to yield two important chemicals from cellulose-based biomass as shown in Scheme 2. The catalytic results revealed that the catalyst activity depends on the structural components of the carbons. In GLY oxidation, the most active catalyst has the lowest carboxylic acid content (Birch-derived carbon). In LA hydrogenation, a higher content of aliphatic structure is found to enhance the stability; therefore, the catalyst (Spruce catalyst). The structure of the carbons does not affect the selectivity of both selected model reactions significantly. The two reactions were performed in water under mild conditions (70–80°C) and revealed a strong support structure. In glycerol, a beneficial effect of carboxylic groups was observed; AuPt supported Birch to be the most effective catalyst. On the other hand, levulinic acid hydrogenation shows a significant feature that rules out the overall catalytic activity, which offers the catalyst's resistance to poisoning. The authors affirmed that aliphatic/aromatic structures with the highest carbon content of which the oxygen is

mainly of ether type appeared to be the most effective support. The AuPt supported on Spruce the most active catalyst. Indeed, the catalyst retained its activity after four catalytic cycles. The used AuPt/Spruce was filtered and, without any further purification, added to a new LA solution. AAS analysis on the filtrate after each reaction excluded the presence of metal leaching [148]. In another study, heterogeneous catalysts were prepared by supporting the homogeneous catalysts iron-phthalocyanines on AC [149]. Different chemicals and thermal treatments, including oxidation in the gas and liquid phases, were used to modify the commercial Noritz carbon surface. Other catalysts were obtained and tested in cis-pinane oxidation with tert-butyl hydroperoxide, at room temperature and atmospheric pressure. The primary product of the reaction was 2-pinane hydroperoxide (77% selectivity at 91% conversion). Interestingly, the carbon surface chemistry effect was observed: supports with very high or very low oxygen content exhibit low activity, whereas for supports with intermediate oxygen contents, a good correlation between the number of phenols and lactones and catalytic activity was observed [149]. Recent findings revealed that; the acetylation of glycerol was performed t over dodecatungstophosphoric acid (PW) supported on AC [150]. The catalysts were prepared by impregnation followed by thermal treatment at 403K. Afterwards, esterification of glycerol with acetic acid was carried out over a series of PW immobilized on activated carbon (3.5–6.5 wt.%). It was observed that the sample with 4.9 wt.% of PW showed the highest catalytic activity. In contrast, higher loading of heteropolyacid on the AC decreased the catalytic activity due to some restraint effect within the AC porous system. The reaction products were monoacetate, diacetin, and triacetin (Scheme 2). optimum values of selectivity to diacetin were observed with all catalysts. The catalytic stability of the sample with higher catalytic activity was evaluated after multiple batches of runs. After the third batch, a stabilization of the catalytic activity was observed [150]. On the other hand, Triacetin (TA) is used

in cellulosic polymers' plasticizer as a solvent. TA and DA are valuable bio-additives for liquid

fuels. These esters can also be used as emulsifiers. Monoacetin (MA) and Diacetin (DA) are the

main components in preparing polyesters and cryogenics.
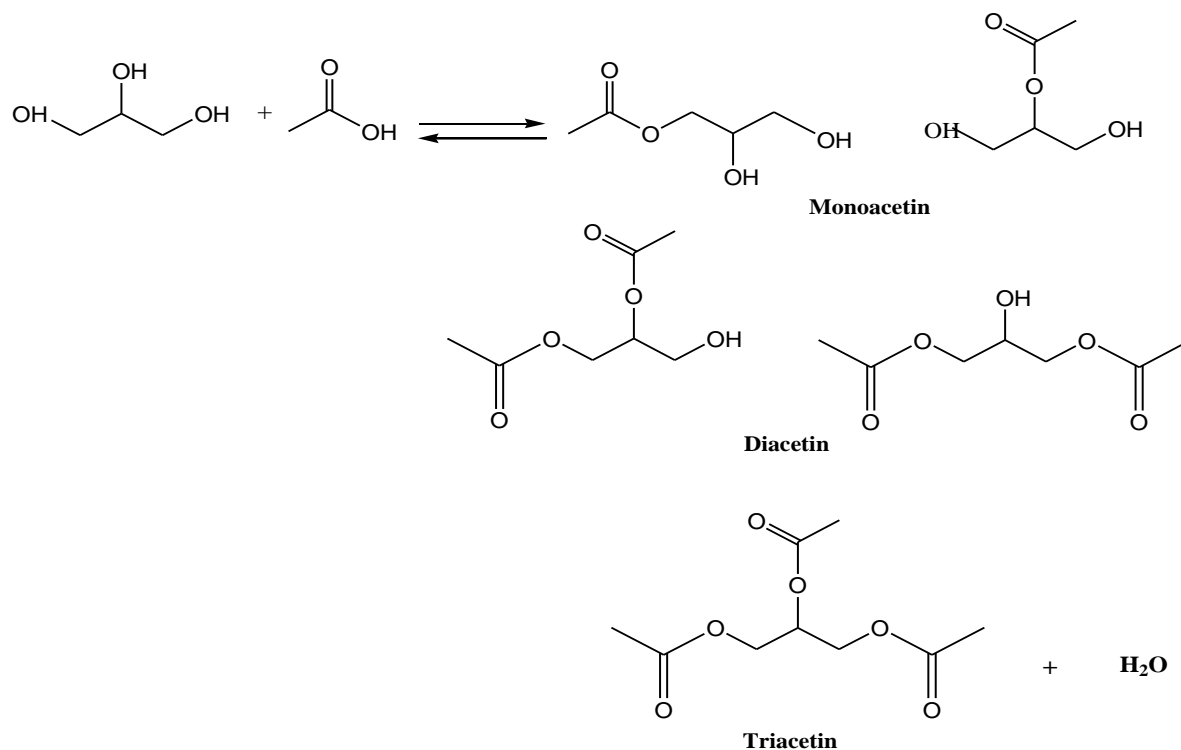
Scheme 2: Esterification of glycerol with acetic acid.

Usually, Triacetin (TA) is successfully employed as a fuel additive to enhance the fuel quality

and as a moisturizer in the Cosmetic Industry [151]. Furthermore, AC with varying textural and

chemical surface characteristics was synthesized from waste biomass and low-rank coals. These

AC were used as a host matrix for cobalt species, varying the preparation and modification

methods by Tsyntsarski and his co-workers [152]. The synthetic activated carbon (SAC),

prepared by thermochemical treatment of a mixture of coal tar pitch and furfural with HNO_3, is

characterised by a relatively high amount of mesopores, promoting the deposition of uniform,

accessible, and highly active in methanol decomposition cobalt species. A similar preparation procedure was used but in H_2SO_4, which led to the highly disordered carbon structure formation. The treatment of this material above 1447 K fully releases the surface functional groups that favour the formation of highly dispersed and relatively high active cobalt particles. As a result, block off accessible reactant molecules, thus decreases their catalytic activity. Despite the indispensable role of carbon paramagnetic centres and oxygen functionalities on the modified cobalt catalytic activity, especially at low temperatures, the dominant effect of carbon texture over the surface functionalities was considered [152]. The expensive PGM catalysts have led to an increasing effort to explore other metals and use AC as support. A good example is illustrated in the hydrogenation of biomass-derived ethyl levulinate to form γ-valerolactone and bimetallic Fe-Ni; this has been extensively used as catalysts support for AC [153]. Other examples are hydrogenation of toluene over Ni/AC catalysts [154], isopropyl alcohol dehydrogenation to acetone over Pt, Cu-Pt and Cu supported on AC catalysts, etc. [155].

From the various reports, it can be concluded that the properties (the particle size distribution, morphology and the attrition resistance of the carbon material) of the synthesized carbonaceous materials are influenced by the choice and nature of starting material used for the production [163]. The porosity influences reactants' access to the active supported metal particles which in turn influence the catalytic performance [153-155,163-164]. In particular oxygen-containing surface groups of the activated carbon support, the surface chemistry influences the catalyst's preparation and the resulting activity. Acidic oxygen functional groups (carboxyl or lactone groups) reduce the carbon's hydrophobicity and increase surface accessibility to the aqueous metal precursor solution. Accordingly, the catalyst must be developed individually for the different applications according to the desired requirements based on empirical and theoretical knowledge [97]. The

catalytic performance of various activated carbons as either catalysts/supports are summarized in table 5

Table 5: Summary of the catalytic performance of various activated carbons as either catalysts/supports

Nature of synthesis	Catalytic systems	Operating parameters	Properties	Brief notes	Ref
Methanol production from biomass syngas	Cu/ZnO/AC	The reaction temperature and pressure 533 K and 60–80 bar were used	$433.2 m^2/g$ (SSA)	The catalyst of methanol synthesis was prepared by Impregnation method of Cu and Zn with activated carbon as a supported catalyst. The result showed that the catalytic activity of Cu/ZnO/Activated-Carbon catalysts through impregnation method was still (1.12% yield methanol) compared	[156]

				to commercial AC (0.2 % yield methanol)	
Catalytic deoxygenation of waste cooking oil (WCO) over acid–base bifunctional catalysts supported on activated carbon (AC) was investigated.	NiLa/AC, NiCe/AC, NiW/AC, NiFe/AC, NiZn/AC NiMn/AC.	3wt% of catalyst loading, 623K reaction temperature, 2 h reaction time under inert conditions were operating parameters	NiLa/AC(336m2/g), NiCe/AC(380m2/g), NiW/AC($130m^2$/g), NiFe/AC($441m^2$/g), NiZn/AC ($446m^2$/g) NiMn/AC($486m^2$/g). Note: specific surface areas are in bracket	Notably, NiZn/AC exhibited the highest hydrocarbon yield at 92%. This indicates that the presence of Ni–O–Zn, along with the presence of acid–base sites in NiZn/AC, was very useful in the production of green liquid fuels. The catalyst was reused for 6 consecutive runs in deoxygenation reaction	[157]
Ketalization of glycerol for the	AC-Carbon Catalyst	Molar ratio of 1 : 8 : 8 (glycerol :	The porous carbon obtained was with a	The catalytic activity of the AC studied for the ketalization of	[158]

| production of solketal as a fuel-additive | was obtained from Zea mays L. cob | acetone : methanol), 1 hour, temperature =50°C, catalyst amount – 5 wt% (w.r.t. glycerol) | specific surface area of 13.901 m^2 g^{-1} and a total pore volume of 0.011 cm^3 g^{-1} . | glycerol lead to maximum glycerol conversion of 72.12% under optimum conditions. The activity of the AC-Carbon Catalyst also did not change appreciably for 3 consecutive batch reaction sequences | |
| Application of activated carbon derived catalyst in methane dry reforming reaction. | Ni/CAC & Ni/ PAC | The reactant gas mixture (CO_2 and CH_4) was purged with nitrogen as carrier gas at flow rate of 50 ml/min and gas hourly space velocity of 30,000 mL h^{-1}g^{-1} over the | **I. PAC (prepared by physical activation)** BET surface area (m^2 /g) =480 | The AC materials were upgraded to supported nickel catalysts (Ni/CAC and Ni/ PAC) and applied in methane dry reforming reaction. Ni/CAC showed higher CH4 conversion (43 %), while Ni/PAC was more suitable for CO_2 | [159] |

| | | reduced catalyst. | Total pore volume (cm^3/g) = 0.26

 Average pore size (nm)= 4.4

 II.   **CAC (prepared by chemical activation)**
 BET surface area (m^2/g) =750
 Total pore volume (cm^3/g) =0.37
 Average pore size (nm)= 4.8 | conversion (31 %). The results showed that both CAC and PAC produced by microwave vacuum pyrolysis of PKS are promising catalyst support materials. | |

Synthesis of biodiesel from waste cooking palm oil	CaO/AC catalyst	T= 313K t=8 h residence time, 295 mm packed bed height and 25 : 1 methanol/oil molar ratio.	Not reported	The CaO/AC catalyst was found significant in the transesterification reaction with the highest conversion of 94%. The catalyst was as well found stable after 4 consecutive runs.	[160]
Transesterification of Dimethyl Carbonate with Propyl Alcohol	K$_2$CO$_3$/AC	Time= 5 h, Temperature= 360 K, n(propyl alcohol)/n(DMC)=3, catalyst dosage=5%	Not reported	The catalyst had an excellent catalytic performance when K$_2$CO$_3$-loading was 15% and catalyst dosage was about 5%. The favorable reaction time was about 5 h, and the lifetime test showed that K$_2$CO$_3$/AC catalyst maintained	[161]

				its catalytic activity during stability tests	
Production of biodiesel using a packed bed membrane reactor	KOH/AC catalyst	The highest conversion of palm oil to biodiesel in the reactor was obtained at 343K with 157.04 g catalyst per unit volume of the reactor and 0.21 cm/s cross flow circulation velocity.	BET surface area= 214.46 m^2/g, Pore volume=0.174 cm^3/g, Micro pore volume= 0.114 cm^3/g, Average pore width= 3.06 nm	The catalytic activity remains 89.3% of the fresh catalyst when the activated carbon supported catalyst is employed for the third time. The decay in catalyst activity could be due to the leaching of active sites to the reaction media. Leaching of the active phase to the alcoholic phase can be attributed to the bond breaking and formation of K^+ and CH_3O	[162]
Production of phenol-	AC & lignite char	The AC and LC were mixed	AC:	The highest bio-oil yield when AC was	[119]

| rich bio-oil via pyrolysis of Palm Kernel Shell over activated carbon (AC) and lignite char (LC) catalysts | (LC) as catalyst | with biomass at different ratios and pyrolysis in a temperature range of 400-600 °C. | BET surface area=707.65 m^2/g

Pore volume= 0.39 cm^3/g

Average pore width (nm)=2.21nm

Lignite char(LC):

BET surface area=93.83 m^2/g

Pore volume= 0.074 cm^3/g

Average pore width (nm)=3.16nm | used as catalyst was 54.73 wt.% at **400 °C** and at PKS:AC ratio of 3:0.5 while the highest bio-oil yield of 60.01 wt.% was achieved at **500 °C** and at PKS:LC ratio of 3:1 | |

7.0 Green Chemistry, Sustainable Development Goals (SDGs) and Circular Economy via bio-derived activated carbon (AC) catalyst

Activated carbon, one of the sustainable products obtainable from renewable sources, has excellent potential to achieve greenness and sustainability through biorefinery. Herein, we represented this

in figure 5, while further details are stressed in figures 6, 7,8, 9 & 10 respectively. The United Nations' (UN) Sustainable Development Goals (SDGs), adopted by 193 UN Member States in September 2015, sets out to achieve several objectives of relevance to the utilisation of resources and waste management [165].
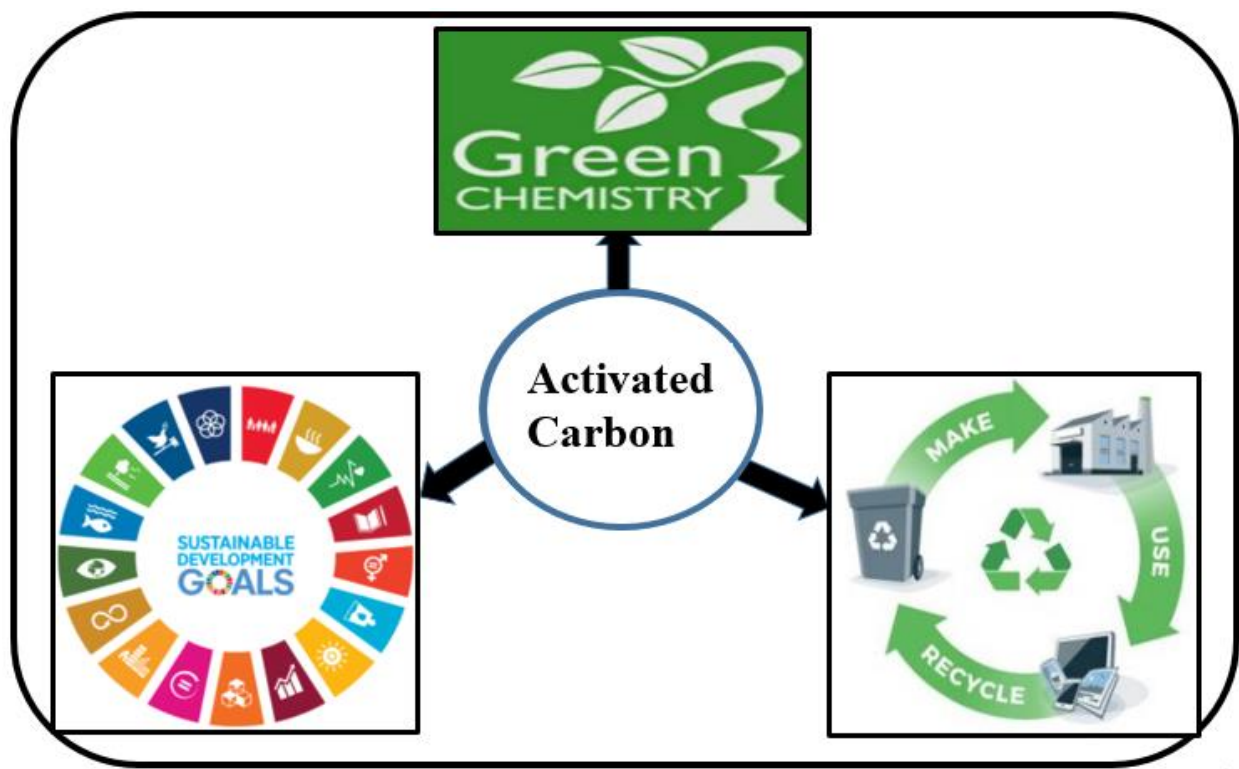

Figure 5: Feasibility and alignment of bio-derived activated carbon with Green chemistry's principles, sustainability, and circular economy. The synthesis of activated carbon from renewable feedstocks/ waste streams conforms with the United Nations (UN) Sustainable Development Goals (SDGs) (#7=affordable and clean energy, #13= climate action & #15=life on land) while circular economy operates in 3Rs (R=Reduce, R=Reuse & R=Recycle).

These resources could directly or indirectly promote sustainable waste and resource management [13-14]. The so-called 3R's Principles: ''Reduce, Reuse, and Recycle' has been used to describe a circular economy which mainly emerges through these three-principal courses of "actions". The

circular economy also elaborately described "as a regenerative system in which resource input and waste, emission, and energy leakage are minimised by slowing, closing, and narrowing material and energy loops" [166]. Indisputably, it has been enhanced by the bio-economy sector's contribution, since sustainable bio-economy represents the renewable segment of the circular economy [10,167-168]. Therefore, numerous value-added products can be obtained by harnessing these readily available alternatives as bio-based feedstocks [169-172]. To this end, a circular economy is considered an approach to address the "make, use, dispose syndrome" of the traditional linear economy. Moreover, in a circular economy, resources are used to maximize the value imbedded in them while in use, then recover and regenerate products and materials after service life [14,167]. Simultaneously, if employed to harness biomass's full potential, it will help reduce our environment's detrimental impacts due to continuous industrial revolution [173,174]. Bio-Based Economy refers to the innovation in biomass effectively utilized to sustainably make bio-based products such as chemicals, materials, fuel, and energy. The merits of shifting the renewable feedstocks afford us an opportunity for innovation and a chance to take advantage of material supply by nature to perform exquisitely selective chemistry. Petrochemical feedstocks provide very simple hydrocarbons, which chemists have learned to make more complex. On the other hand, natural feedstocks are inherently different and believed to be less harmful. They are complex molecules, and chemists are racing to develop elegant methods to transform them into useful products [175] efficiently. However, one way to increase our economy's circularity is to create business models around secondary materials, promoted by supporting policies [43]. Therefore, we recognize an approach to transform biomass/ waste streams to various value-added products as a practical guide to the policy resonance and implementation. Thus, production of bio-derived activated carbon, other value-added products, and biofuels through the utilization of

lignocellulosic biomass/agro-wastes and other waste streams are undoubtedly conformed with the Green Chemistry principles (i.e. principles **#1, #2, #6, #7 & #9**), circular economy and sustainability with the green light of eco-friendliness towards environmental protection. Utilization of bio-derived activated carbon in the catalytic system is promising and could play a key role in developing a sustainable future and is of immense importance to realize the United Nations Sustainable Development Goals.

7.1 Bio-derived Activated Carbon and Green chemistry's principles

According to figure 6, feasibility and alignment of bio-derived activated carbon with Green chemistry's principles are stressed below.
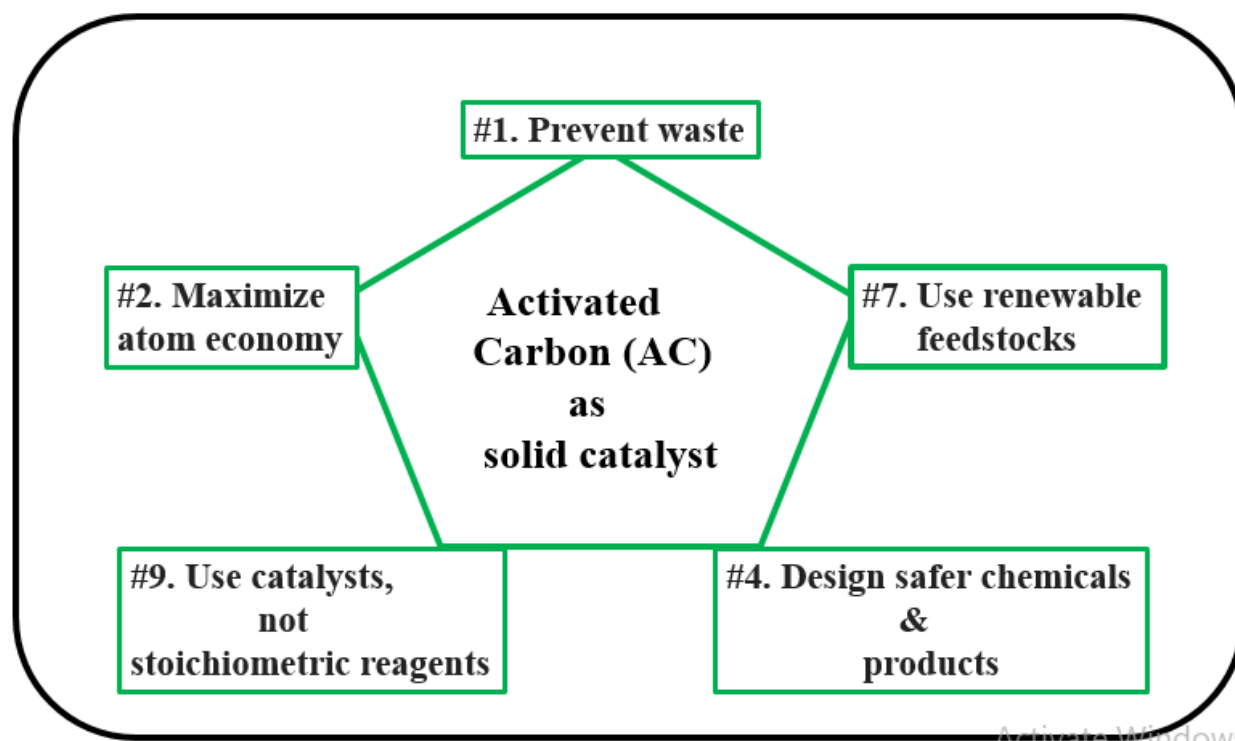

Figure 6: Feasibility and alignment of bio-derived activated carbon with Green chemistry's principles. Green chemistry principles (#1, #2, 4#, 7#, and 9#) are aligned with the synthesis and utilization of bio-derived activated carbons as solid catalysts compared with traditional homogeneous catalytic systems.

#1. Prevent waste: This employs the design chemical syntheses to prevent waste. Leave no waste to treat or clean up- waste generated from various sources that could constitute a nuisance to the environment. It is employed in the synthesis of activated carbon-a solid catalyst. Moreover, during synthesis, various value-added products are obtained, which are eventually useful as precursors for producing different materials or end products. **#2. Maximize the atom economy**: Design syntheses so that the final product contains the maximum proportion of the starting materials: waste few or no atoms. About two decades ago, the catalytic system was crucial to attaining a better atom economy to create an alternative route/pathway by lowering energy transition and activation barriers [99,175]. Therefore, as the intrinsic efficiency of a balanced chemical reaction, the atomic economy could also dictate the design for chemical synthesis in green chemistry and the idea of 100% atomic economy would be an ultimate goal for the eco-friendly process [90,97,149,157]. **#4. Design safer chemicals and products**: Design chemical products that are fully effective yet have little or no toxicity. **#7. Use renewable feedstocks**: Use starting materials (also known as feedstocks or precursors) that are renewable and recyclable rather than non-renewable ones. The feedstock utilization plays a vital role in the design and manufacturing of the products. Some defective or less useful products are considered for reuse or recovery by contributing to the prevention of wastes or waste management in general [167]. **#9. Use catalysts, not stoichiometric reagents**: Catalyzed reactions minimize waste by using catalytic reactions. Catalysis can depict the energy requirement by reducing the enormous amount of debris. Catalysts are useful in small amounts and can carry out a single reaction many times. They are preferable to stoichiometric reagents, which are used in excess and carry out a reaction only once [99,106,176]. The requirements for the shift towards the energy and resource sustainability are illustrated in

figure 7, according to the reported works [14,167,174]. Using activated carbon as either catalysts or supports has justified a few principles of Green Chemistry. The activated carbon catalysts reported here were used severally, no issues of corrosion issues with the equipment were reported, unlike homogeneous counterparts.
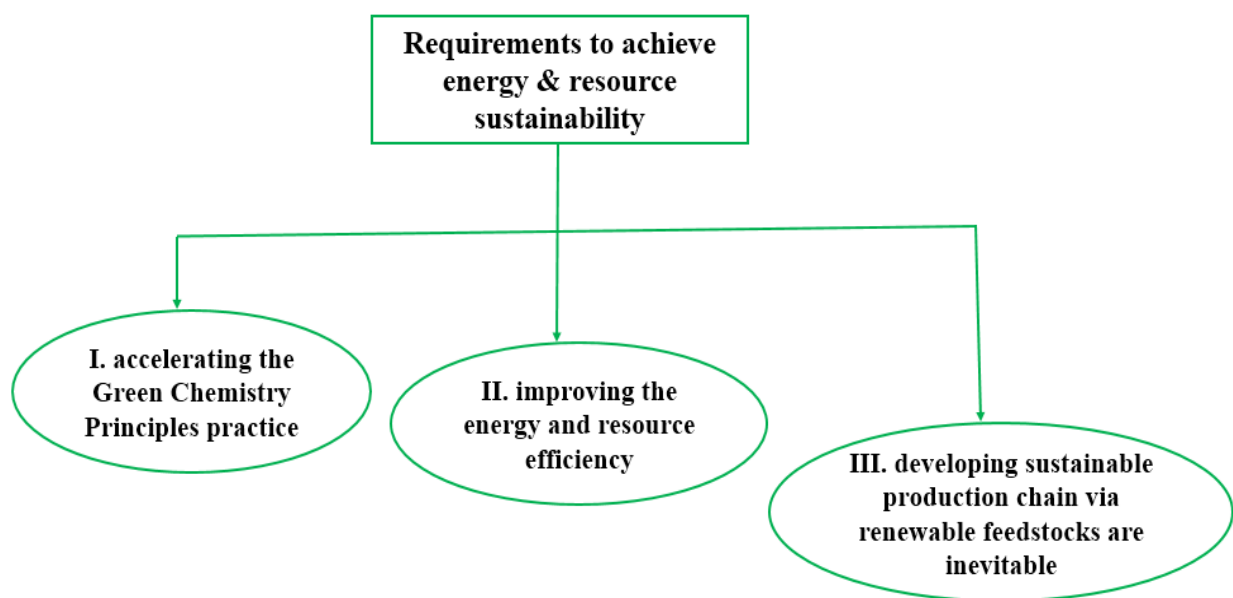

Figure 7: Requirements to achieve energy & resource sustainability

7.2 Utilization of Bio-derived Activated Carbon towards Sustainability

The landmark Paris Agreement, signed in April 2016 by 175 Member States, aims to mitigate climate change, accelerate and intensify actions and investments needed for a sustainable, low-carbon future. The integral part of this agreement is the need to strengthen the global response to limit global temperatures from rising no more than 2 degrees Celsius above pre-industrial levels and to ensure further efforts are made to limit the rise to 1.5 degrees Celsius. The Paris Agreement requires parties to identify their "intended nationally determined contributions" (INDCs). Progress on the Paris Agreement will be tracked every five years through a global stocktaking exercise. A comparison would be made of global greenhouse gas emission levels in 2025 and 2030 resulting

from the implementation of the intended nationally determined contributions (INDCs and under other scenarios (GtCO$_2$ eq/yr.) [159]. To achieve better sustainability, the effective utilization and adequate management of renewable feedstocks/streams of wastes should be adopted to pursue this course. This will promote environmentally benign and socially acceptable strategies to preserve the environment from detrimental effects of non-renewable resources [15,21]. Although Sustainable Development was transformed into a UN plan only in 2015, the process of integrating the economy, environment, and ethics has existed for about three decades in history. The primary element in the concept of sustainable development is an interrelationship between people, resources, and environment, and development [177]. Taking this into account, the SDGs (#7=affordable & clean energy, #13= climate action, and #15=life on land), production of activated carbon from bio-renewable resources possesses the ability to achieve sustainability (see figure 8). From the available materials reviewed, we represent the anticipated benefits of using biomass waste as raw material to synthesize carbon-based catalysts in biorefinery in figure 9. Therefore, the transformation of these huge wastes (agro-wastes, municipal wastes, industrial wastes etc.) generated to renewable fuels and value-added products (e.g. activated carbon) is an added advantage in sustainability.
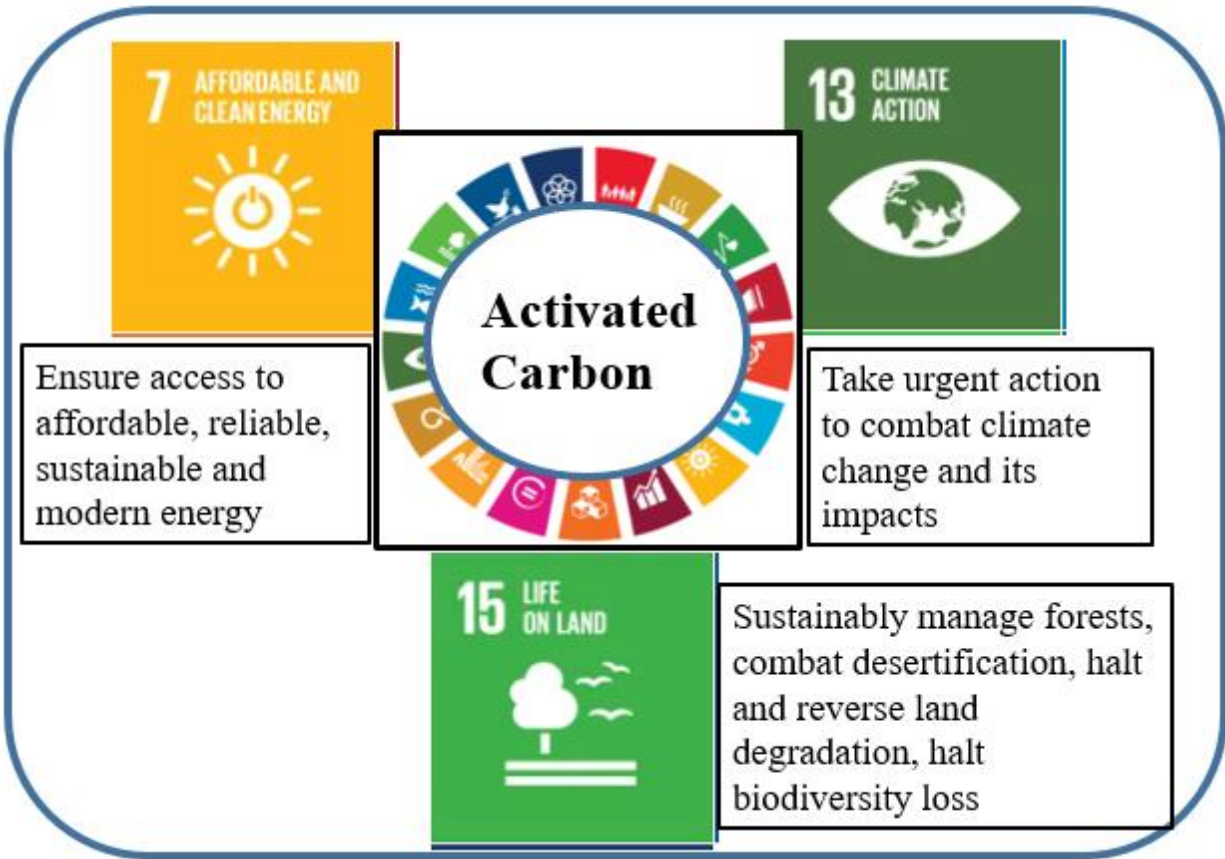
Figure 8: Feasibility and alignment of bio-derived activated carbon with a circular economy. Transforming waste streams into activated carbon and subsequent use as solid catalysts/supports possess characteristic features of 3Rs (R=Reduce, R=Reuse & R=Recycle) of circular economy and achieving sustainability via biorefinery.
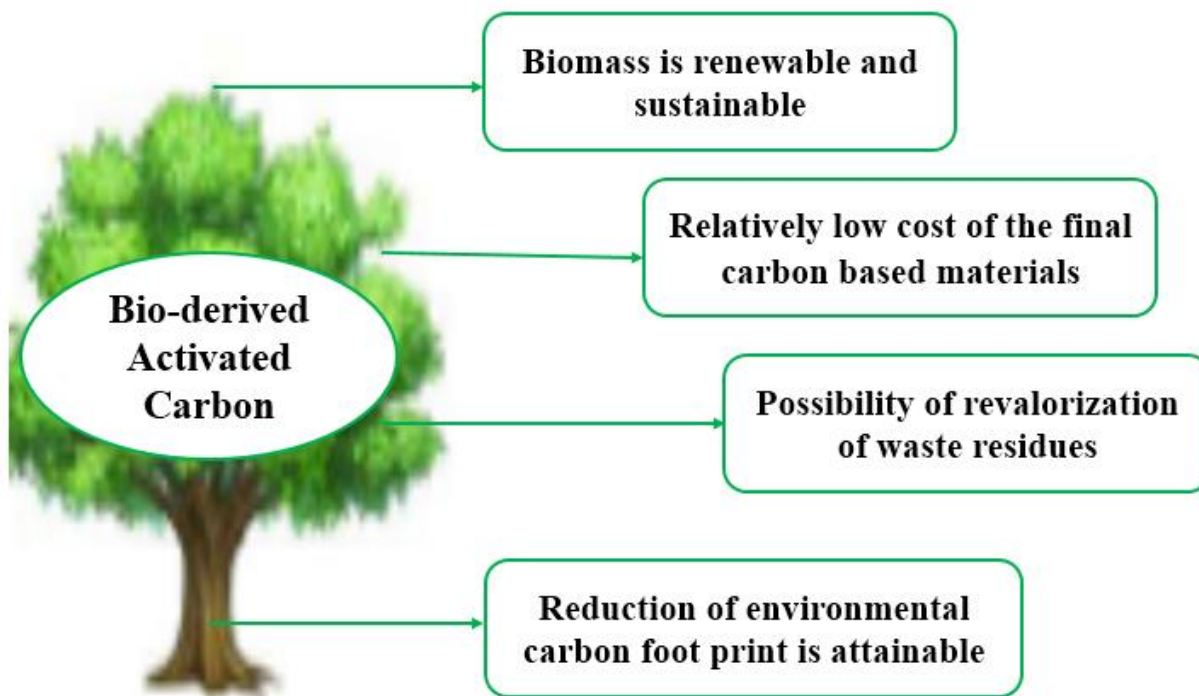
Figure 9: Advantages of using biomass waste as raw material for the synthesis of carbon-based catalysts in biorefinery

7.3 Utilization of Bio-derived Activated Carbon towards a circular economy

For sustainability reasons, and to address detrimental environmental impact, there is increasing interest in exploiting bio-based feedstocks/waste streams to develop value-added products (VAPs) and production of bioenergy (biofuels). This will effectively drive the sustainability focus to the waste hierarchy threshold (reduce, reuse, recycle) [21,166-167]. To tackle the pressing problems in sustainable production, the activated carbon derived from renewable sources have been reported with a heterogeneous catalytic system in biorefinery [128,136-137]. A traditional linear approach to production and consumption (take, make, dispose of a syndrome) is unsustainable. Therefore, new circular techniques (make, use, return) are growing in influence and importance with global economic growth and urbanization (see figure 10). Recent advances in catalysis are particularly

vital to the circular pursuit of "keeping the molecules in play" [178]. The cases examined in this study revealed that catalysts are also obtainable from biomass and can be reused and recycled in contrast with homogeneous systems [127-128,134,137]. Therefore, these catalytic systems with bio-derived activated carbon would be applicable as a pivotal technology to develop more sustainable biorefinery protocols [178]. Development of effective catalytic methodologies and processes will enable biomass-derived precursors to produce other useful products such as personal care products, high-performance materials, and pharmaceutical ingredients. More sustainable manufacturing processes are required to realize the circular economy. There is an imperative need to develop new catalysts and catalytic processes across various chemicals and materials supply chains [167,178]. Therefore, new circular approaches (make, use, return) are growing in influence and importance with global economic growth and urbanization (see figure 10). Thus, the circular economy and sustainability concepts through synthesis and application of activated carbon underpin improved selectivity/efficiency, application, and end-of-life (recycling or degradation) overusing non-renewable resources.
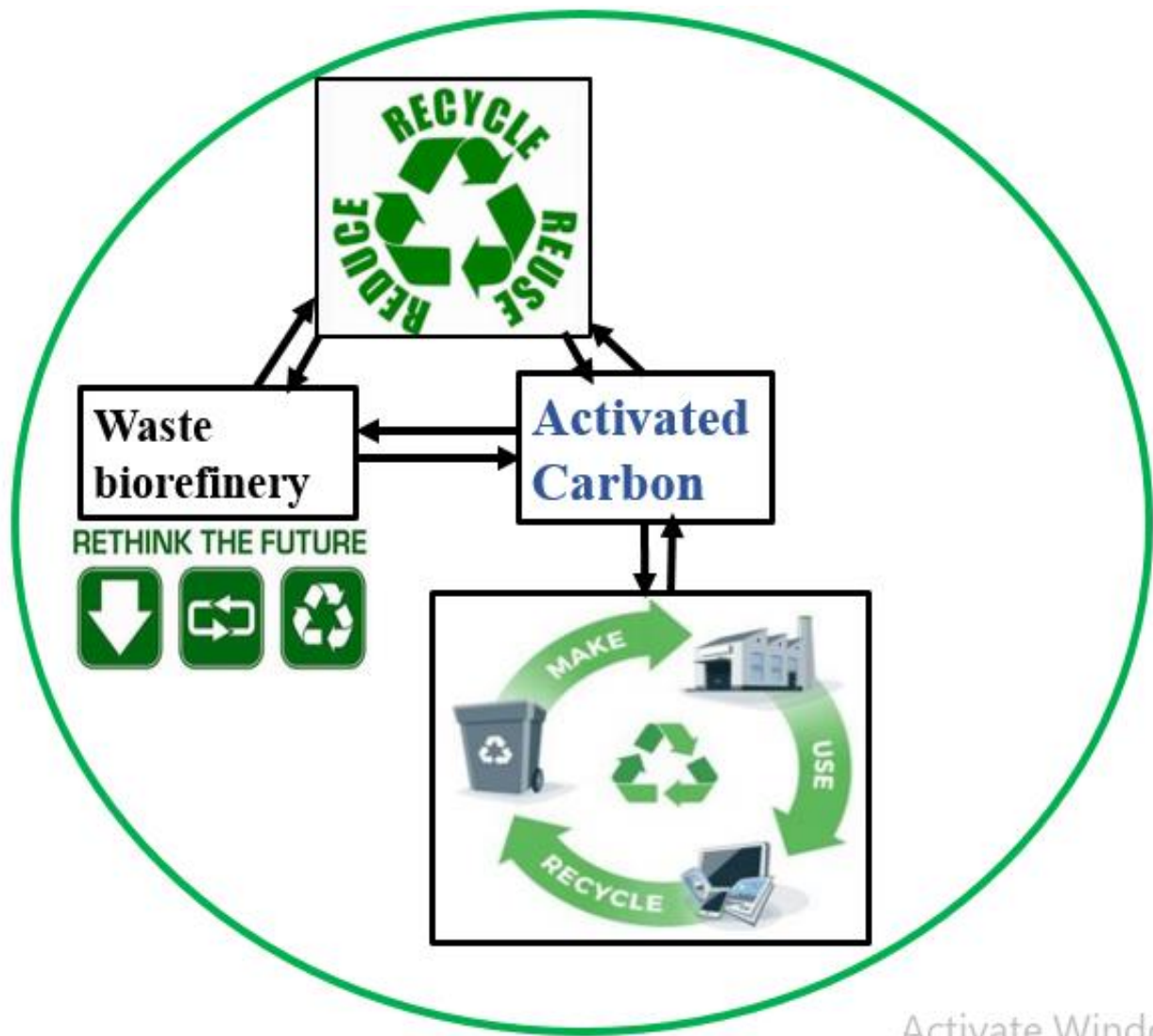
Figure 10: Feasibility and alignment of bio-derived activated carbon with a circular economy. Transforming waste streams into activated carbon and subsequent use as solid catalysts/supports possess characteristic features of 3Rs (R=Reduce, R=Reuse & R=Recycle) of circular economy and as well towards achieving sustainability via biorefinery.

8.0 Evaluation of solid catalysts, recent progress & our opinions on the utilization of activated carbon in biorefinery

Bio-based activated carbon (AC) has received significant attention because of its eco-friendliness and efficiency. The significance and potential of the AC as solid catalyst/supports in transformation of renewable feedstock to value-added/biofuels have shown a very promising glimpse of what lies ahead in the future of biorefinery. Several materials (such as activated carbon, nanoparticles, zeolites, CNTs, etc.) have been employed as an efficient catalyst for the synthesis of liquid hydrocarbon fuels and value-added chemicals (VACs). However, there are existing challenges in the cost-effectiveness and sustainability of some previously reported materials in catalytic systems. There have been countless reports about the development of catalytic systems in terms of selectivity and activity. At the same time, consideration for sustainable synthesis seems to be left out of the plan or receive little or no attention. Economical and sustainability of the catalytic materials must be considered as the world is yearning for a sustainable and friendly ecosystem. In recent time, many researchers are developing solid catalysts. AC as catalyst or supports loaded on other materials is more effective and efficient to realize sustainable synthesis and a shift towards biorefinery via circular economy. Besides, these bio-derived catalytic materials are reusable in several cycles which is advantageous in industrial applications. Therefore, a need to intensify development on technologies for a safer, more sustainable, and low-cost synthesis of bio-derived carbonaceous materials from eco-friendly and renewable precursors is inevitable. This act is profitable, productive, beneficial to realize sustainable synthesis and even be of great value to waste reduction.

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9.0 Conclusions and Perspectives

The approach of green chemistry through its principles #7 (use of renewable feedstocks) and #9 (catalysis) corroborate the feasibility of circular economy and SDGs by leveraging activated carbon potential value-added product, and biofuels from biomass/waste streams. A renewable source has been a vital key to realizing eco-friendly and sustainable production useful materials/products. The promising features of activated carbon produced from lignocellulosic biomass wastes by physical or chemical activation steps made an indispensable material for the sustainable economy. Using waste biomasses for the synthesis of activated carbon, value-added products, and biofuels will address the problems associated with environmental degradation and the economic viability is of utmost importance as it will ease the burden of allocating funds to waste management sector annually. As reviewed herein, activated carbon either as solid catalysts or supports for biomass transformation is a vital alternative to traditional homogeneous catalytic systems. Throughout the reported works discussed in this review, activated carbon has demonstrated high potential for catalytic applications due to porous structure, chemical stability and good catalytic activity. Notably, a key feature of activated carbon is its various functional groups relevant to specific catalytic reactions. The type and the distribution of these functional groups can be modified by oxidative agents, either in gas-phase or in solution by thermal treatments preparation protocol. Notably, biomass-derived catalyst, however, is often difficult to distinguish from catalysts of other carbon sources. In specificity, its properties (pore size and surface groups) determine their optimum performance. Owing to the unique shape- and porosity-controlled properties, these carbon materials could offer strong, active phase-support interactions, leading to unusual catalytic activities and selectivity in biomass upgrading. A key advantage of such synthesis processes is that the raw material (biomass) is abundant and renewable. Porous

carbons have been developed and used as a heterogeneous catalyst in fine chemical and biofuels synthesis as a sustainable and economical alternative to other solid catalysts. From the concepts of this work, the production of AC, VAPs, and biofuels may be considered a pathway towards achieving a fully circular economy to salvage our environment from further degradation. Considering the sustainable nature of these materials and its expected growth in the application, new methods and procedures continue to appear in academia and industry. Therefore, it is anticipated this approach would synthesize such value-added materials more sustainable, eco-friendly, and economically viable. More efforts are targeted toward a full-scale utilization of biomass. However, studies on how to resolve the waste washing solutions generated and the sustainability of the chemical agents are scarce. Therefore, this needs to be given more consideration apart from precursors employed from a sustainable and renewable point of view. The long-term stability of the catalysts (*e.g.*, leaching of components) and the appropriate disposal of the spent catalysts are also excellent. Besides being a sustainable material and approach, more studies need to be done mostly on its economic analysis or life-cycle assessment for more efficient management. Thus, activated carbon remains an excellent candidate, exciting and challenging research subject that requires a deep commitment to being fully sustainable. In summary, there are many promising potentials in the use of biomass in catalysis as demonstrated by the bio-derived activated carbon (AC), therefore activated carbon holds a prominent role to facilitate the development of modern biorefinery. Using these agro-wastes, municipal wastes, industrial wastes etc. will also increase the revenues and reduce wastes.

Conflicts of interest

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

Credit authorship contribution statement

Aderemi Timothy Adeleye: Conceptualization, Methodology, Investigation, Writing- Original draft preparation, Resources & Software. **Amos Adeleke Akande:** Writing - review & editing, Supervision & Funding acquisition. **Chuks Kenneth Odoh:** Investigation, Writing - review & editing, Validation, Supervision. **Monday Philip:** Writing - review & editing. **Timothy Tizhe Fidelis:** Writing - review & editing. **Pigweh Isa Amos**: Writing - review & editing. **Oluwakemi Oluwabunmi Banjoko:** Visualization & Validation.

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The Editor-in-Chief

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30th December, 2020.

Dear Sir,

Point-by-Point Responses to the Reviewers' Comments on our Manuscript
(**Ms. Ref. No.: JIEC-D-20-02551**).

Thank you for your efficient work in processing our manuscript entitled "**Efficient synthesis of bio-based activated carbon (AC) for catalytic systems: A green and sustainable approach**" (**Ms. Ref. No.: JIEC-D-20-02551**). Those comments are all valuable and very helpful for revising and improving our paper, as well as the important guide to us. We have read and studied suggestions/comments carefully and have made the correction which we hope to meet with approval. The main corrections in the paper and the response to the reviewers' comments are as follow:

Point-by-point responses to the reviewers' comments are enclosed after the letter for your consideration.

-**Reviewer #2**: The manuscript describes a GOOD work AND is well presented. Authors need following points to be included before reconsideration.

1. Abstract should contain some quantitative information also.

Response to Reviewer #2

1. Thanks for your suggestion, we have added quantitative information in the abstract with red colour.

2. English must be improved.

Response to Reviewer #2: Thank you very much sir. This comment has been addressed

3. Novelty of the work be established.

Response to Reviewer #2: We have done this in line with your recommendation. Sir, we recently introduced new section (section 8.0: **Evaluation of solid catalysts, recent progress & our opinions on the utilization of activated carbon in biorefinery**)

4. All the adsorbents reported be compared in a tabular form to establish the superiority of the work.

Response to Reviewer #2: Thank you very much for the recommendation. This has been responded to in Table 5 (Summary of the catalytic performance of various activated carbons as either catalysts/supports)

-**Reviewer #3**: Bio-based activated carbon (AC) has received significant attention because its eco-friendliness and efficiency. This review presents the significant and potential of the AC as solid catalyst/supports in transformation of renewable feedstock to value-added products/chemicals and renewable biofuels. There are some revisions should be addressed before it can be accepted.

1. Reviewer: The theme of this article should be the efficient synthesis of AC as the title shows, but most of this article is focused on the application of AC in the synthesis of value-added products/chemicals and renewable biofuels.

Response to Reviewer #3: Thank you sir. We have attended to this in section 3.1, equations were provided for systematic synthesis, table 1 was also introduced to buttress the point. Just after the table 1 brief notes were given in the body of work in red colour.

2. Reviewer: As a science review paper, the manuscript should summarize the scientific principles of the references instead of enumerating them when quoting.

     Response to Reviewer #3: Thank you very much for this recommendation. This comment has been addressed. We just introduced table 3 (Characteristic properties and characterization techniques for the novel activated carbon) under section 4.0 **(Characteristic properties and characterization techniques for the novel activated carbon)** & table 5 (Summary of the catalytic performance of various activated carbons as either catalysts/supports) for this purpose (this can be found immediately after section 6.4 in red colour).

3. **Reviewer:** There are too many grammatical errors in the manuscript. The authors must check the manuscript and refine the language carefully.

     Response to Reviewer #3: Thank you very much sir. This comment has been addressed

Dear Sir, all your suggestions have been attempted and effected accordingly in the revised manuscript. We are thankful to you for sparing your precious time to read our manuscript and providing important suggestions on how it can be improved. We hope that our manuscript would be accepted and finally publish in Journal of Industrial and Engineering Chemistry.

Thank you very much.
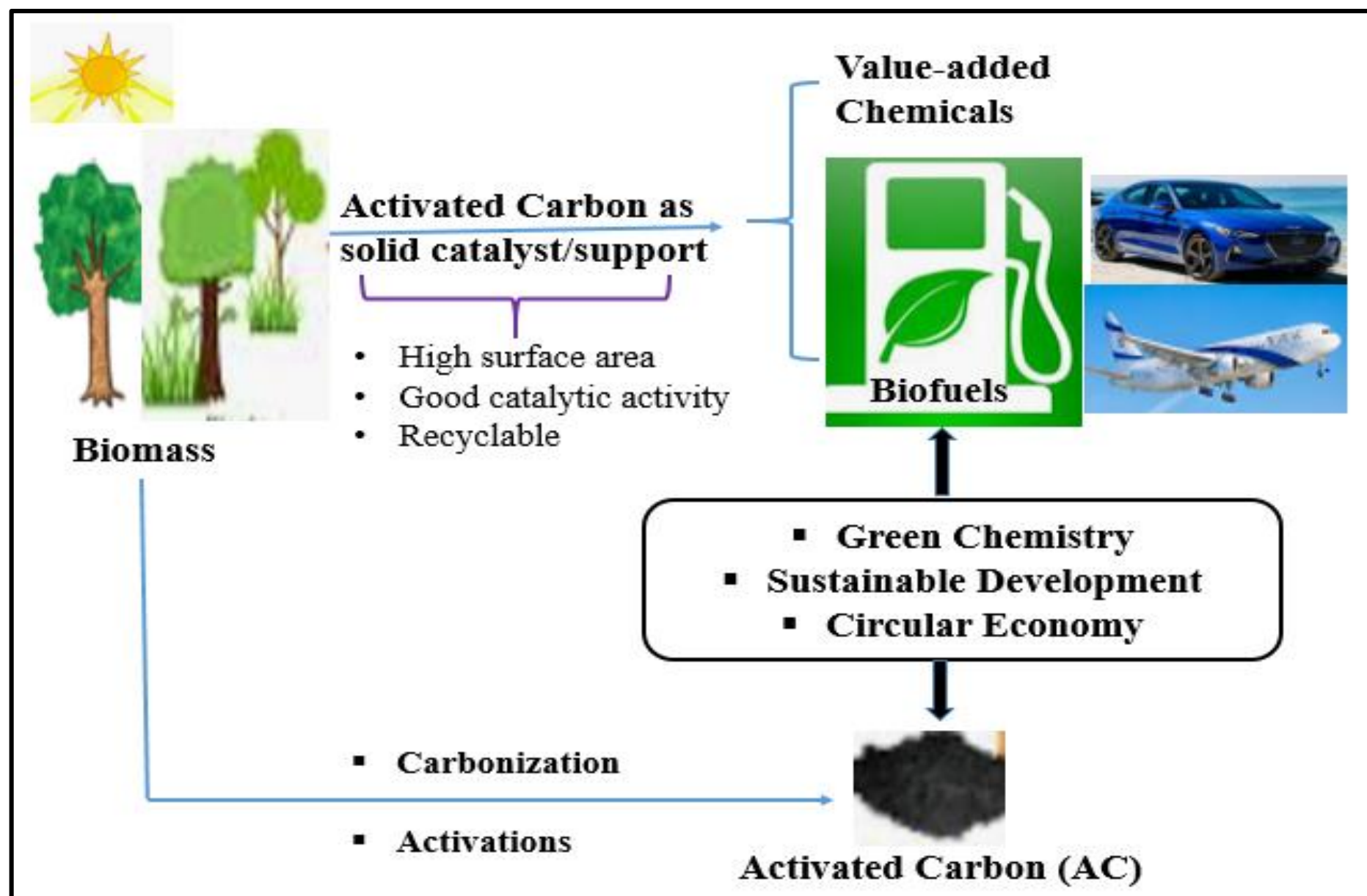
Declaration of interest

The authors declare our interest to publish our manuscript with Journal of Industrial and Engineering Chemistry, Elsevier

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