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Characterization of Activated Carbon Fibre Derived from Palm Empty Fruit Bunch Waste and Cassava Stem for Urea Adsorption

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Abstract: The study of activated carbon fiber derived from agricultural waste has bring a lot of attention to researchers all around the world. Activated carbon fibers generated from cassava stem and empty fruit bunch waste have a high propensity to serve as a dispersing agent where chemical and physical treatments may change the activated carbon surface. The characteristics of activated carbon which is highly porous, has large surface area make it suitable to be used in many applications in the chemical, pharmaceutical and food industries. Hence, the objective of this study is therefore to determine the effect of activation temperature on the empty fruit bunch waste and cassava stem. This study revealed that increasing of BET Surface area, total pore volume and micropore volume influenced by the activation temperature. However, macropore decreasing at temperature above 900 °C lead to less efficiency in adsorption function. More total and fixed carbon, high BET surface area, total pore volume and micropore volume for adsorption, activated carbon fiber with high activation temperature (800 °C) produced. The best chemical agent for impregnation process between acidic and basic also were studied. Thus, the basic which is KOH frequently used by many researchers compared to the other chemical agents as it has been found to be effective in activated carbon production with a narrow pore size distribution and well-developed porosity. In short, the chemical agents and the parameters were important in producing activated carbon fibre derived from agricultural waste.

Keywords: Activated Carbon, Agricultural Waste, Pore Structure

1. Introduction

Activated carbon (AC) defined as a carbonaceous solid and porous material that derived from coal or biomass via thermochemical processes. There are 3 forms of activated carbon; fibres, granular and powdered. Activated carbon fibre (ACF) is a strong, fibre-shaped microporous material with a well-defined porous structure [1]. The characteristics found that it can be used in many applications such as

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gas purification, waste water treatment, decaffeination, gold purification, metal extraction, water purification, medicine and as filters in compressed air. However, the processing cost of activated carbon fibre is very costly when compared to the other activated carbon. The combination of fibre processing cost and activation cost is the reason for the processing cost to be higher [1]. Therefore, palms empty fruit bunch waste and cassava stem have been choosing as a cheap raw material for activated carbon (AC) production, with lower energy consumption. Previous research by [2] stated that empty fruit bunch fibre has been proven to be good absorbent for urea and it will act as control for this study. Hence, this project is carried out based on methodology from [2] by using different material as activated carbon fibre which are from EFB and cassava stem, basically to analyses the adsorption rate of urea. Therefore, it can be used to identify the best AC in urea absorption either EFB or cassava stem can produce activated carbon in absorbing urea. The modified activated carbon fibre from these two different materials will be tested in control sample of urea water mixture to study the absorption performance.

The current study of activated carbon fibre that derived from empty fruit bunch waste and cassava stem didn’t have strong proof as the activated carbon fibre also can be derived from other materials. In this study, the function in adsorbing urea was depend on BET surface area, total pore volume and average pore size for EFB and cassava stem. SLR systematic literature review was used in this project to solve this problem by identifies, evaluates, and synthesizes another studies research that lead to the excellent summary. Through SLR, it can help to have a clear evidence in using cassava stem and EFB for the production of activated carbon.

The objectives of this study are to study the chemical agent that have been use for the EFB and cassava stem for impregnation process. Next, to study the effect of activation temperature on the EFB and Cassava stem from last decades. Lastly, proven the use of EFB and Cassava stem in the production of AC for adsorption the best sources between EFB and cassava stem in producing activated carbon fibre.

2. Literature Review

2.1 Empty fruit bunch fibre (EFB)

According to [2] stated that Oil Palm Empty Fruit Bunch (OPEFB) also known as EFB where it is biomass that can be alternative energy source as it is a by-product of palm-fruit harvesting. Oil Palm Empty Fruit Bunches (OPEFB) are the fresh fruit bunch (FFB) solid residues of oil palm trees that are mainly grown in several countries including Indonesia, Malaysia, Thailand and others [3]. Empty fruit bunch (EFB) is a sort of biomass produced during the process of palm oil processing. Empty fruit bunches (EFB) are what remains of the fresh fruit bunches following removal of the fruit for oil pressing. Empty fruit bunches are a form of mill waste that is of great value to farmers, since it provides nutrients and increases the soil's organic content [5].

2.2 Empty fruit bunch fibre (EFB) properties

Oil palm empty fruit bunch fibre (EFB) has been characterized by tensile power, Young's modulus, break elongation, density, and Etc. The findings recorded are not identical due to variations in the form of oil palm fibre used and also the irregular sectional area that fluctuates along the length of EFB. Hence, some of the sources large range of values reported for the mechanical properties. Additionally, researchers have documented linear stress-strain diagram of EFB tensile action, but there is no comprehensive diagram [6]. Oil Empty fruit bunch fibre (EFB) major chemical components are cellulose 65 percent, lignin 19 percent, and ash 2 percent. Tensile, flexural, and shear behaviour of materials are the most significant properties of structural elements but authors could not find any study or paper on the shear properties of EFB composites [6].

2.3 Cassava stem

Global production of cassava uses approximately 19 million hectares of land, including unproductive areas with low precipitation. The biomass of cassava stem may be as much as 50 percent of the root mass, but the role of cassava has so far been ignored in both starch production and energy production. For certain countries, part of cassava stems is either used as a fuel for cooking, although the process of igniting and making flames is longer than other biomass, or reincorporated into the soil with the aid of a mechanical chipping to add organic matter and nutrients. Other cases the excess stems have to be washed from the fields for preparation for the growth of the next season and discarded or burned in the wild, causing pollution and environmental problems [4].

2.4 Number of pore development

Pore size is usually defining as the difference between the two opposite pore walls (diameter of cylindrical pores, slip-shaped pores width). According to the International Union of Pure and Applied Chemistry (IUPAC) stated that there are three groups of pores classification where it is micropores, mesopores and macropores. The size of the adsorption pores is classified into less than 2 nm (<2 nm), around 2–50 nm, and larger than 50 nm (>50 nm) respectively. The pores size gives large impact on the porosity, surface area and give effect to the size of molecules for the diffusion into the solid. So, the appropriate development of pore size is necessary for the production of the activated carbon that also can use in other applications [5]. The pore-size distribution of the activated carbon is the factor determine the adsorption characteristic and it also determine the structural heterogeneity of porous material.

2.5 Adsorption capacity

Adsorption power (or loading) is the amount of adsorbent absorbed by the adsorbent per adsorbent unit mass (or volume). According to the study of development of activated carbon in 2016 stated that adsorption performance is the most important characteristic of an activated carbon where it is also influencing the surface characteristic of activated carbon and its pore development. The adsorption capacity increases lead to increasing micro porosity by using physical activation [5]. Based on the research by [2] stated that the adsorption of urea decreased with increasing of acid impregnation ratio where when the acid-to-fibre ratio rose from 0.75 to 4, the equilibrium amount of urea adsorbed on activated carbon fibre (ACF) decreased from 877.907 to 134.098 mg g^{-1} [2].

3. Methodology

This section of the thesis will discuss the searching process based on three objectives to retrieve relevant articles and study selection. This research review is composed according to PRISMA which stands for Preferred Reporting Items for reporting in systematic reviews and Meta-Analyses is used in this systematic review study. This study selection including a flow diagram with the numbers of studies are given with reasons for each stage. This strategy involves identification, evaluation, studies selection and explanation of exclusion. Subsequently, the papers included are listed after the method of exclusion where this process eliminates 50 articles and have left 50 articles for quality evaluation.

3.1 Study selection

It shows the recorded number of studies that have been identified from bibliographic sources and it also shows how the process of selection. From the flow diagram at Figure 1, we can see that a total of potential articles obtained are 100 articles through manual searching from ProQuest, Scopus, Science Direct, Google Scholars, Web of Science and Google engine Search. After adjusting for the basis of title and abstract, 70 remained. 30 studies were discarded because after reviewing the abstract, it did not meet the criteria and it is also duplicates. The parameters to be summarized are that it includes the characteristics of pore form, chemical reagent usage, surface area and overall pore volume. The 70 reports reviewed in depth relate to the analysis of the manuscript and the implementation of the inclusion criterion. It seems that 20 studies did not meet the inclusion requirements mentioned above. The studies left are 50 studies since they meet the requirements for inclusion by reviewing the related publications, the features that are being researched and by checking the studies that quoted those articles. No unpublished related reports have been obtained. The results and discussion section presents data and analysis of the study. This section can be organized based on the stated objectives, the chronological timeline, different case groupings, different experimental configurations, or any logical order as deemed appropriate.

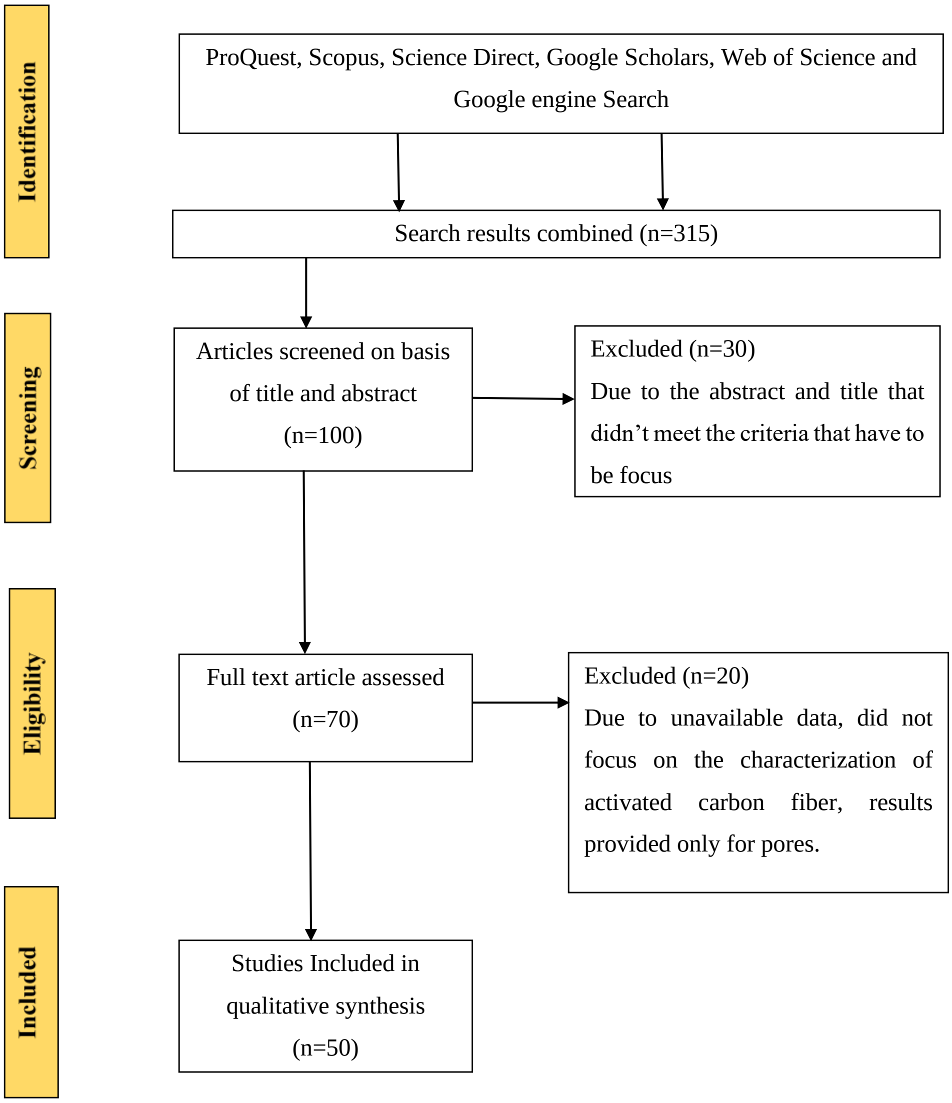

Figure 1: Flowchart of search strategy and selection process [6]

This study used two main searching techniques, namely advanced searching on the selected database. Second, manual searching on four main data base which were Scopus, Science Direct, Google Scholars and Google engine search. This study also used three main techniques where it is handpicking, backward tracking and forward tracking.

4. Results and Discussion

4.1 Limitations in finding the past decades research of EFB and cassava stem

From the past decade's studies showed that activated carbon derived from EFB is available to be find as discussed in chapter 3 also it has been discussed by some authors but for cassava stem, the discussion on cassava is limited. Numerous academic papers dedicated to the characterization of synthesis and the application of novel precursors to activated carbon processing can now be identified as other researcher have been collect the data of precursors in produced the activated carbon shows in Table 1 but the keywords of cassava stem are none. The study on cassava that can available is cassava leaves and cassava peels which still come from new journal around 2009 to 2020 years.

Table 1: Summary of the lignocellulosics precursors used as precursors for the synthesis of activated carbon [10]

Precursor	Reference
Cassava root	[38]
Cassava peels	[39]
Empty fruit bunch	[40]
Empty fruit bunch	[41]
Oil palm empty fruit bunch (EFB)	[31]
Oil palm empty fruit bunch (EFB)	[42]
Waste tea	[43]

4.2 Chemical activation

Chemical activation is known as wet oxidation where it is required catalyst to be impregnated into raw materials and followed by washing method to produce activated carbon fibre. The final stage in chemical activation is washing step where the activated carbon is depending on the chemical reagents used in the preparation, is essentially washed with acid or alkali and then washed with water. On top of that, washing step in chemical activation is important step to develop porosity as the washing step remove chemical components and in the carbon structure, the porosity of the resulting activated carbon is essentially filled by the chemicals [30].

4.2.1 Five common chemical agents used in chemical activation

The chemical agents or chemical catalyst that usually used in chemical activation were $ZnCL_2$, H_3PO_4, H_2SO_4, NaOH and KOH has reported by [26] and [30]. From 50 journals, 5 studied were presented in Table 2 to prove that chemical agents that usually used for empty fruit bunch (EFB) were $ZnCl_2$, H_3PO_4, H_2SO_4, NaOH and KOH.

Table 2: Chemical agents used to be impregnated into EFB

Precursor	**Chemical agent**	**Reference**
Palm Oil Empty Fruit Bunch	NaOH	[29]
Oil Palm Empty Fruit Bunch	H_3PO_4	[25]
Empty Fruit Bunch	$ZnCL_2$	[30]
Coconut husk	KOH	[26]
Palm Empty Fruit Bunch Waste	H_2SO_4	[32]

Whereas the percentage chemical agents used for impregnation process of EFB have been studied from 50 journals and presented into the pie chart (Figure 2). About 34 % refer to 20 articles have use KOH while 30 % refer to 15 articles use $ZnCl_2$ followed by the use of H_2SO_4, NaOH and H_3PO_4 where strong base gets a lot of attentions in production of activated carbon derived from empty fruit bunch waste. The reason for basic agent being choose frequently stated in Figure 2.

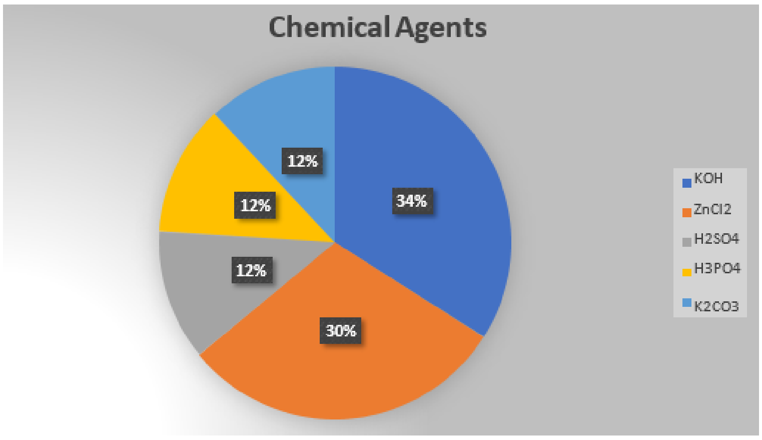

Figure 2: Percent of the most common chemical agents from 2009-2020 [26]

4.3 The effect of basic and acidic used as catalyst to be impregnated with EFB in porosity development of surface area, total pore volume and pores size

One of the major advantages of chemical activation is producing higher development of porosity where it has been listed in Table 4.4 for the use of acidic and basic as chemical agent that also function as catalyst to help in developing porosity. The disadvantage in chemical agent is usually about cost and the need to perform washing step to remove the chemical agent [33].

Table 3: Maximum adsorption capacity of ACs prepared from agricultural wastes by chemical activation under the optimum preparation conditions [10]

Acidic	Reference	Basic	Reference
Acid accumulation modifies the volume of the micropore during phosphoric acid activation.	[27]	Narrow and large porosity is created by the alkali agent	[26]
The largest level of micro porosity acid inhibit production facilitates the creation of mesopores and macropores	[28]	The alkali agent does not stimulate the growth of mesopores and micropores	[26]
Zinc chloride provides a model effect and generates a structured micropore function	[26]	Potassium hydroxide material plays a key function in the creation of pores	[26]

From the review about the effect using acidic and basic chemical agents for porosity development shows the important words such as micropore, mesopores and macropores. These terms are the classification of pore sizes of activated carbon that categorized into three types which is micropore (pore size < 2nm), mesopores (2nm < pore size < 50nm), and macropores (pore size> 50nm). According to

Yahya et al., 2015 stated that in angstrom unit, the pore size has been defined as micropore (8-100 Å), mesopores (100-500 Å), and macropores (500-20,000 Å).

From the Table 4 represented that acidic such as $ZnCl_2$ and H_3PO_4 efficient in producing micropores, mesopores and also macropores compared to the basic agent that doesn't produce mesopores and micropores. The use of alkali agent which is KOH has been found that it is effective in activated carbon production with a narrow pore size distribution and well developed-porosity. So, both acidic and basic chemical agent could lead to pores development but the data from pie chart shows higher percentage of researcher use KOH more compared to $ZnCl_2$ and H_3PO_4. This is because KOH is most eco-friendly [34] and can produce narrow and wide micro porosity compared to $ZnCl_2$ and H_3PO_4. The production of activated carbon derived from empty fruit bunch waste using $ZnCl_2$ cannot be used in food and pharmaceutical industry. Another study reported that $ZnCl_2$ could give environmental impact.

4.4 Results of surface area, total pore volume and average pore size

There are several journals stated that related trends in BET surface area and porosity were observed for the development of activated bio char at the raising of temperature [35] supported by [36]. In addition, activation temperature and KOH impregnation play important role to determine the yield of activated EFB waste.

4.4.1 Effect of activation temperature on empty fruit bunch waste in producing activated carbon for effective adsorption

In addition to providing a strong relationship between activation temperature and surface area, total pore volume and average pore size, the activation temperature was checked and analysed in order to achieve the optimal activated carbon yield. The sample of EFB was dried and filtered overnight at 120 ℃ in an oven during the manufacturing process of activated carbon fibre extracted from empty fruit bunch waste. After that, the sample was activated at 400 ℃ in a furnace for 45 min. As the study focus on the activation temperature, the 45 min retention time will be fixed. The activation temperature was then verified in the 400 °C to 800 °C range shown in Table 4 and Table 5.

Table 4: BET surface area and pore volume of palm shell activated carbon [40]

Sample	BET surface area (m^2/gram)	Total pore volume (cm^3/g)	Micropore volume (cm^3/g)
5AC	521.5	0.215	0.217
6AC	631	0.314	0.288
7AC	905	0.569	0.449
CAC	860	0.55	0.48

Table 5: BET surface area, total pore volume, micropore volume and average pore diameter of activated EFB under various activation temperatures [37]

Sample	BET surface area (m^2/g)	Total pore volume (cm^3/g)	Average pore diameter (cm^3/g)
AB 400	90.65	0.02	0.007

AB 600	580.47	0.1	0.06
AB 800	920.67	0.24	0.17

According to the Table 4 and Table 5 resulted on the BET surface area, total pore volume, and micropore volume with different activation temperature. As can be seen from the study, both results reported that where the growing temperature of activation from 500 °C to 700 °C and 400 °C to 800 °C contributes to an increase in the surface area of BET, total volume of pore and volume of micropore. In the meanwhile, the further expansion of the activation temperature from 400 °C to 800 °C decreased the pore diameter substantially from 83.66 to 20.86 A. The decreasing of the average pore diameter when activation temperature increase is due to the development of micropore volume. The increasing of activation temperature enhances and widening the existing pores and create new porosities. The pore will be widening upon activation with CO_2 flow when activation temperatures increasing resulting mesopores volume to be increase too. In addition, this parameter provides an excellent medium for ion exchange capability during the adsorption process, and the BET activated carbon surface area indicates that it is ideal for adsorption for all data produced, as the findings were within the range of 400-1400 m^2/g of commercial activated carbon.

5. Conclusion

In recent years, there are increasing number of researchers that interested and studies on this kind of preview where activated carbon can be prepared from biomass and agricultural or natural materials where it is widely used but this study limit for cassava stem whereas EFB have most journal to be reviewed. So, this study most focus on derivation of activated carbon for effective adsorption. Other than that, chemical agent contributed important function in porosity development where basic KOH get the most reviewed and use during impregnation process compared to acidic. The biggest reason for the use of basic chemical agent to prevent environmental effect as KOH is eco-friendlier. Moreover, for EFB to have effective adsorption must have highest BET Surface area, total pore volume and micropore volume and this parameter depend only on activation temperature. The highest temperature, the highest the surface area, higher total pore volume and higher micropore volume for adsorption. In short, EFB have been proven to be the strong raw material for the development of activated carbon fibre for adsorption because, with the aid of KOH, it has reached the higher BET surface area, higher micropores in producing narrow and formed porosities.

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