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# *MELALEUCA CAJUPUTI* LEAVES AS A RAW MATERIAL FOR PREPARATION OF ACTIVATED CARBON

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**Abstract:** In this study, activated carbon was prepared from *Melaleuca cajuputi* leaves. The activation included both physical and chemical processes, with phosphoric acid ($H_3PO_4$) as a dehydrating agent, followed by thermal treatment at 500°C for 45 minutes. The activated carbon obtained was characterised by surface area (BET), FESEM (images), elemental analysis (EDX), and Fourier transform infrared spectroscopy (FTIR). The sample subjected to carbonisation without chemical activation showed a low surface area of 24 $m^2/g$ at 500°C. The treatment of samples with chemicals showed improvements in surface area and other characteristics. Chemical activation using phosphoric acid seemed to be a very efficient method and with surface area improvements reaching as high as 127 $m^2/g$. Therefore, *M. cajuputi* leaves can be used to produce activated carbon with good surface properties and used in the treatment of environmental waste due to its sustainability.

Keywords: Sustainability, activated carbon, *Melaleuca cajuputi* leaves, FTIR, percentage yield.

## Introduction

Activated carbon is a black solid material like powdered or granular, charcoal (Abdullah *et al.,* 2001), processed from carbonaceous materials such as plants, coal, graphite, and peat. It possesses a large surface area, a well-developed porous structure, and a rich surface group (Jankowska, 1991; Xu *et al.,* 2014). Activated carbon is widely used due to its high adsorption capacity and large internal surface area (Houache *et al.,* 2009; Stavropoulos *et al.,* 2009). Activated carbon can be derived from various materials containing carbon, including graphite and peat or materials with minor carbon content like cellulosic or lignocellulose, which includes wood waste, fruit shells and stones (olive, peach, apricot, etc.), cane bagasse, rice husk, and olive cake (Deiana *et al.,* 1998; Hayashi *et al.,* 2002).

The activation of carbon was done either via a direct activation of the raw material or two-phase methods, involving an initial carbonisation preceding the activation process (Heidarinejad *et al.,* 2020). The activation processes were either physical or chemical. A physical activation involves the partial gasification of carbonised material in the presence of steam or carbon, leading to the removal of reactive carbons. Meanwhile, a chemical activation involves impregnating the material with a chemical compound such as $H_3PO_4$, followed by thermal treatment. This research paper found the best approach to be chemical activation because of its high pore volume, large surface area, low activation temperature, percent yield, and observable absorption capacity (Tran *et al.,* 2017). The properties of the activated carbon depend on factors and conditions during processing such as temperature, pressure, reactor setup, and the activating agent used (Foong *et al.,* 2022).

The global dependence on non-renewable materials such as lignite, crude oil, and coal

for the production of porous materials presents significant ecological challenges. This reliance contributes to raised costs in food stocks and is associated with environmental degradation (Haimour *et al.,* 2006). Therefore, research into other sources and techniques for the production of activated carbon that are accessible, inexpensive, and adhere to environmental standards is vital. Gelam (*Melaleuca cajuputi*), a biomass rich in lignocellulose content, serves as a viable candidate for activated carbon production (Habibah *et al.,* 2022). *M. cajuputi,* a tree species from the family "Myr-taceae", is a ubiquitous tree found on the east coast of Peninsular Malaysia and is particularly abundant in Terengganu (Abdullah *et al.,* 2018). This species mainly grows in humid, hot climates, which is found in Malaysia. *M. cajuputi* can also adapt to unfavourable conditions, particularly in areas where flooding is intense. The plant exhibits exceptional growth and yields in peat swamp regions, where many other crops and forest trees struggle to survive.

*M. cajuputi* plantations produce wood, charcoal, honey harvested from bees that nest in the branches, and essential oil extracted from leaves (Glesen & Sari, 2018). Over the years, the leaf extract has been widely used in many medicinal applications, in Southeast Asia (Barbosa *et al.,* 2013). The production of *M. cajuputi* timber generates waste in the form of wood chips and leaves, which becomes organic waste (Anggarini & Muzaidi, 2021). This solid waste transforms eco-friendly activated carbon, which can be further enhanced or tailor made for a diverse range of applications, most notably adsorption (Singh *et al.,* 2020b). The quantity of solid waste produced by *M. cajuputi* plantations has the potential to pose environmental contamination risks (Ibrahim *et al.,* 2023). Thus, a simple, eco-friendly method is necessary to convert the leaves into valuable products.

*M. cajuputi* leaves are being used for many applications such as the extraction of essential oils (Glesen & Sari, 2018), herbicides (Kuah *et al.,* 2019), and wood preservation (Patramurti *et al.,* 2020). However, the authors of this research paper have found that no prior research has been done on utilising *M. cajuputi* leaves to produce activated carbon. Therefore, this study aims to prepare activated carbon from *M. cajuputi* leaves. The physicochemical characteristics of this material and the production methods used to obtain products with highly developed porous structures are examined (Deiana *et al.,* 2009). In addition, the product is a cost-effective and an efficient substitute for commercial carbon, presenting opportunities for environmental waste remediation.

## Materials and Methods

### *Experimental Setup*

The experimental work of preparation and characterisation of activated carbon was conducted at Besut Tembila Campus Laboratory, Universiti Sultan Zainal Abidin, 21300 Kuala Nerus, Terengganu, Malaysia.

### *Preparation of the Sample*

*M. cajuputi* leaves were collected from Hutan Simpan Jambu Bangkok, Setiu, Terengganu. The leaves consist of alternate leathery leaf blades that are greyish green, lance-shaped, measuring between 3 and 12.5 cm in length and between 1.1 and 3.75 cm in width with pronounced longitudinal veins, as shown in Figure 1. These samples were washed with water to remove mud or dust before being dried in the sun and cut into 2 mm-thick slices. The sample was further washed with distilled water and dried in an oven at 110°C for a day (Anisuzzaman *et al.,* 2015).

The dried cartels were subjected to carbonisation and activation. In physical activation, 20 g of the sample was carbonised in a furnace at a temperature of more than 500°C at the rate of 15°C/minute under nitrogen flow, followed by $CO_2$ activation with a similar treatment (Sekirifa *et al.,* 2013). In chemical activation, the sample was impregnated with phosphoric acid (85 wt%). The impregnation was performed using 100 ml of different concentrations of phosphoric acid ($H_3PO_4$) (2M and 4M) as a dehydrating agent. The chemical

activation was achieved by impregnating 20 g of the sample with $H_3PO_4$ concentrations and shaking it in water for three days at 80°C at a speed of 70 rpm (Anisuzzaman *et al.,* 2015).

After the impregnation, the leaf samples were dried at 110°C for 2 hours and introduced into the furnace for carbonisation. The temperature was initially set at 200°C, each sample underwent semi-carbonisation for 20 minutes. Subsequently, the temperature was adjusted to 500°C and the sample was activated for 45 minutes, as outlined by Anisuzzaman *et al.* (2015). Following the carbonisation process, the activated carbon was subjected to multiple washes with hot distilled water to remove any residual acids. This washing continued until a stable pH was consistently observed (Zięzio *et al.,* 2020). The percentage yield of the activated carbon can be expressed by the equation below:

$$\text{Yield of AC \%} = \frac{wf}{wi} \times 100\% \quad (1)$$

where *wi* is the initial weight of the impregnated sample and *wf* is the final weight obtained after the activation process.

### *Characterisation of Activated Carbon*

The physical and chemical properties of activated carbon were characterised as follows. Fourier transform infrared spectroscopy (FITR) was conducted to analyse the functional group, providing insight into the chemical structure of the produced activated carbon. The surface area was measured using Quanta chrome auto-sort Gas sorption analyser Brunauer-Emmett Teller (BET).

Elemental analysis and morphological structure was examined by Energy Dispersive X-ray microanalysis (EDX) (Oxford Instrument X-Max 80) to study the content percentage of some elements originating from the adsorbent and Field Emission Scanning Microscopy (FESEM) (Carl Zeiss Gemini SEM 500) to analyse the morphological structure.

## Results and Discussion

### *Percentage of Yield*

The findings presented in Table 1 highlight the influence of various precursor parameters on the yield of activated carbon. Anisuzzaman *et al.* (2015) demonstrated that the quantity of $H_3PO_4$ employed significantly affected the evaluation of the resulting activated carbon. The yields obtained ranged between 33% and 38.7%. It was observed that increasing the concentration of $H_3PO_4$ led to an enhancement in the breakdown *of M. cajuputi* leaves. This finding indicates the importance of $H_3PO_4$ in facilitating the carbonisation process. Notably, these results are consistent with those reported by Reffas *et al.* (2010) and Martín-González *et al.* (2013)

Figure 1: *Melaleuca cajuputi* leaves

in their investigations on the production of activated carbon from lignocellulose waste treated with $H_3PO_4$ before carbonisation.

The lowest percentage was observed at MC-AC1 with 33% while MC-AC3 showed the highest percentage yield of 38.7%. Thus, an increase in the amount of acid improved the substrate. This might speed up the process and lower the yield (Olawale, 2012). In this regard, phosphoric acid serves as a dehydrating agent in the production of activated carbon yield (Liu *et al.,* 2009). It also promotes the redistribution of biopolymers and favours the conversion of aliphatic to aromatic molecules, as well as dehydration, consequently increasing the activated carbon yield (Yakout & Sharaf-el-Deen, 2012).

### *Surface Area BET*

The surface area of the activated carbons derived from *M. cajuputi* leaves is presented in Table 1. Physically activated carbon has demonstrated an increased surface area of approximately 24 $m^2/g$ when subjected to an activation temperature of 500°C, surpassing the findings of Houache *et al.* (2008), who achieved a surface area of only 1.0 $m^2/g$ through activation temperatures ranging between 400 and 500°C. The surface area increased in chemically activated carbon. A slight decrease in surface area was noted with an increase in $H_3PO_4$, which is consistent with research by Abdullah *et al.* (2001). It is significant to note that lower concentrations of the impregnation agent compared to those that other scholars have used (Molina-Sabio *et al.,* 2003) led to the development of porous adsorbents. The surface area variation after chemical treatment demonstrates numerous and related impacts of various sample modifications.

The pH of activated carbon can be used to define the pH of suspension carbon in distilled water (Abdullah *et al.,* 2001). As observed from Table 1, the pH value ranged between 4 and 5, indicating acidity. This pH was achieved through multiple washes of the carbonised activated carbon with distilled water to eliminate acids and other residues. However, the activated carbon pH had an impact on the surface chemistry and adsorption of different adsorbates that are sensitive to pH fluctuations (Anisuzzaman *et al.,* 2015).

### *FESEM Morphology*

Figure 2 displays the FESEM images of *M. cajuputi* leaf-activated carbon prepared using 0M, 2M, and 4M concentrations at an activation temperature of 500°C. The pores from the starting material were observed in both micrographs. After impregnating the material with $H_3PO_4$, the surface textures changed (Houache *et al.,* 2008). The texture was denser in Figures 2 (b) and (c), followed by pores of varying sizes and shapes. This indicates the presence of large-sized clear pores and cracks (Kan *et al.,* 2016). Figures 2 (a), (b), and (c) demonstrate how the shape of the pore changes from MC-AC2 to MC-AC3 as the impregnation ratio increases. Elemental dispersive X-ray microanalysis (EDX) was conducted to ascertain the composition of elements derived from the *M. cajuputi* leaf adsorbent. The following elements were identified and are presented in Table 2: C, O, P, S, K, and Ca.

These elements comprise primarily of carbon (70-78%), oxygen (17-28%), phosphorus (~2-12%), and calcium (~0.1-2%) . The existence of other elements is because of the nature of the initial organic substance. As observed, the

Table 1: Phosphoric acid concentration, percentage of yield, pH, and surface area

| Sample | $H_3PO_4$ Concentration | Percentage of Yield (%) | pH | BET Surface Area ($m^2/g$) |
|---|---|---|---|---|
| MC-AC1 | 0M | 33 | 5.2 | 24 |
| MC-AC2 | 2M | 37.5 | 4.42 | 127 |
| MC-AC3 | 4M | 38.7 | 4.51 | 34 |

material under study has a common composition for organic carbon compounds. This affirms the precursor since it is an organic material. The changes in the elemental content agree with the concentration of oxygen surface functional groups. The high oxygen contents confirm the presence of surface oxygen groups, which are greater than P, S, K, Ca, and Al due to surface oxidation resulting from the action of $H_3PO_4$ during activation. Similar results were reported by Isinkaralar *et al.* (2023) using pinecone black for carbon production.

Most lignocellulose (plant dry matter) such as *M. cajuputi* leaves have high-quality carbon and oxygen contents for producing activated carbons averaging 40-60% and 40-45%, respectively (Alcañiz-Monge *et al.,* 2022; Isinkaralar *et al.,* 2023). Moreover, it is essential

Table 2: Elemental analysis of MC-AC1, MC-AC2, and MC-AC3 (mass %)

| Element | MC-AC1 | MC-AC2 | MC-AC3 |
|---|---|---|---|
| C (%) | 78.05 | 69.04 | 70.07 |
| O (%) | 17.79 | 23.35 | 19.96 |
| P (%) | 1.54 | 6.99 | 9.31 |
| S (%) | 0.58 | - | - |
| K (%) | 0.80 | - | - |
| Ca (%) | 1.23 | 0.62 | - |
| Al (%) | - | - | 0.66 |

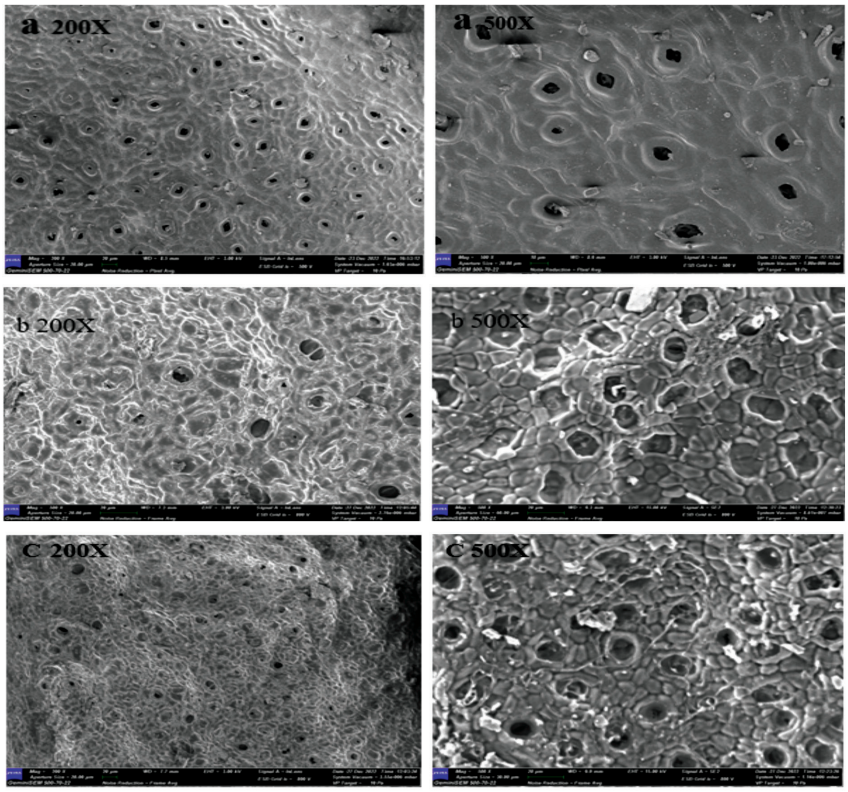


Figure 2: FESEM images of (a) MC-AC1, (b) MC-AC2, and (c) MC-AC3 at 200X and 500X magnification

to note that the amounts of sulphur, potassium, calcium, and aluminium decreased, as indicated in Table 2. The primary source of phosphorus was from the material and might be due to impregnation with $H_3PO_4$.

### *Fourier Transform Infrared Spectroscopy (FT-IR)*

Figure 3 shows Fourier transform infrared spectroscopy (FT-IR) presenting the surface functional groups of the produced activated carbon. FITR examination gives data on chemical structure and breaks down and discovers the oxidation formation of activated carbon. Figure 3 demonstrates the spectra of the *M. cajuputi* leaves activated carbon, MC-AC1 (physical), MC-AC2, and MC-AC3 (chemical) respectively. As observed from Figure 3, the three activated carbons possess almost similar spectra, only MC-AC1 has a slight difference. These indicate that the samples have similar functional groups and patterns.

Varying concentrations of $H_3PO_4$ do not cause any significant changes in the spectra for MC-AC2 and MC-AC3 (Figure 2). These correspond with the study of Anisuzzaman *et al.* (2015). The activated carbon made from cattail leaves produced similar spectrum readings (Shi *et al.,* 2010; Ren *et al.,* 2011). The broad band at 3,500 $cm^{-1}$ originates from the O-H stretching vibration of the hydrosol group of *M. cajuputi* leaves. The peak at 3,000 $cm^{-1}$ is caused by stretching the C-H bond in the benzene ring (Stankovich *et al.,* 2006). Stretching vibrations at 1,400 to 1,650 $cm^{-1}$ are attributed to C=C bonding. The presence of this functional group suggests that the material has been aromatised and that a carbonyl-containing group has formed (Guo *et al.,* 2006). The band around 1,000 $cm^{-1}$ corresponds to the C-O group.

In chemical activation using $H_3PO_4$, the chemical activator encourages the depolymerisation, dehydration, and redistribution of constituent biopolymers. This process disintegrates the lignocellulose material after it undergoes pyrolysis (Anisuzzaman *et al.,* 2015). As a result, at low temperatures, it is possible to increase the conversion of aliphatic to aromatic compounds (Jagtoyen *et al.,* 1993). In the spectrum of the chemically activated carbon the band around 800-1,050 $cm^{-1}$ is attributed to the phosphoric-containing group. The peaks at 1,050-900 $cm^{-1}$ can be related to P=O, O-C, and P=OOH bond. Also, the bands at 2,600 and 2,400 $cm^{-1}$ were related to C-H, which corresponds with the study by Gomez-Serrano (Gomez-Serrano *et al.,* 1995).

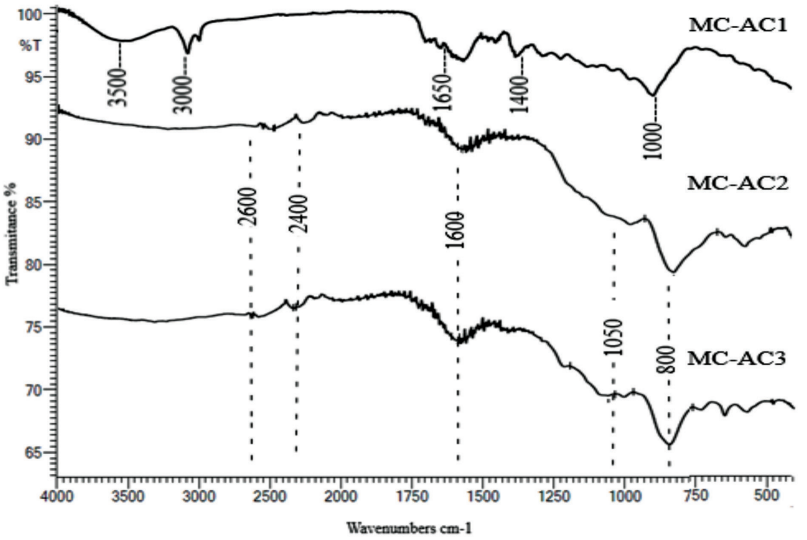


Figure 3: FTIR spectra of the three activated carbons

## Conclusions

*Melaleuca cajuputi* leaves are abundant and can be used as a raw material to produce activated carbon. The physical activation requires the removal of reactive carbons from the carbonised material before activation. By removing the reactive carbon, the fixed carbon content increases to levels comparable to those of other materials used to prepare activated carbon. The final product was produced with a surface area of 24 m²/g. Chemical activation of *M. cajuputi* leaves using phosphoric acid as the activating agent is an effective activation method, as final products with BET surface areas between 34 and 127 m²/g are obtained. The comparison of those activated carbon surface areas prepared by physical and chemical methods showed that the one prepared by chemical activation had larger clear pore connectivity than the one prepared by physical activation. The results indicated that impregnating the material with phosphoric acid (chemical synthesis) is more effective in obtaining activated carbon with high porosity, optimum yields, and a product with a surface area of 127 m²/g. The study has shown that *M. cajuputi* leaves could be used as raw material for the preparation of activated carbon with good surface properties. Further research (additional research) is required to focus and shed more light on other properties of activated carbon.

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## Conflict of Interest Statement

The authors declare that they have no conflict of interest.

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