KOH Activation with Microwave Irradiation and its Effect on the Physical Properties of Orange Peel Activated Carbon

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Abstract. Chemical activation with assisted microwave irradiation was used to produce activated carbon from orange peel waste. The activating agent was potassium hydroxide (KOH) with concentrations of 2 M, 3 M, and 4 M. The microwave irradiation was done for 15 minutes with a 630 Watt output power. KOH concentration affected the physical properties of OP-ACxM. With increasing KOH concentration, the interlayer spacing ($d_{002}$ and $d_{100}$) grew, stack height ($L_c$) increased, and stack width ($L_a$) dropped. The number of pores on the surface of OP-ACxM increased after the chemical activation process. In OP-ACxM, FTIR analysis reveals the presence of O-H, C-H, C=C, C=O, C=O, and C-O.

1. Introduction
Activated carbon (AC) is a common term for carbon materials, which comprises charcoal. AC is applied in water, wastewater, remove the odor, and some heavy metals adsorption. AC can be produced from agricultural waste materials such as coconut shells [1,2], corn stalk [3], oil palm empty fruit bunches [4–6], sago waste [7], pineapple leaf [8], etc. ACs have high adsorptive capabilities because of their fine and porous structure and huge particle surface area [9]. Adsorption capacity, sustainability, and the use of bio-solvent and other residual wastes as adsorbents have all been proven to be competitive with other techniques of removing contaminants [10]. One of the most often utilized industrial adsorbents for purification, separation, and recovery operations is commercial activated carbon[11].

The production of the activated carbon process usually using carbonization and activating the carbonaceous raw materials [12]. Physical activation entails first carbonizing carbonaceous materials to remove volatile stuff, then activating them using activating agents such as CO$_2$, air, steam, or a mixture of these activating agents [13]. Chemical activation involves impregnating the raw material with chemicals such as ZnCl$_2$, KOH, NaOH, and H$_2$SO$_4$, this process was involved heat-treated in an inert atmosphere through a one-step process [14,15]. The application of assisted microwave irradiations on chemical activation and regeneration for the activated carbon preparation process has proved successful [2,16]. Microwave irradiation is generating heat and adsorbed by the sample elements through the dielectric heat process. In this study, activated carbon was prepared from orange peel through pre-carbonization and chemical activation with assisted microwave irradiation. The activated carbon was produced at different KOH concentrations. X-ray diffraction, scanning electron
microscopy, energy dispersive X-ray, and Fourier transform infrared were used to characterize activated carbon.

2. Experimental method

Three major processes have been used to prepare activated carbon from orange peel utilizing potassium hydroxide (KOH) as the activating agent. The three processes are (i) pre-carbonization, (ii) chemical activation, and (iii) microwave irradiation. Cleaning the orange peel of any stuck dirt and cutting it into 5 mm pieces is the first step in the synthesis of activated carbon. In an electric oven, the pieces of orange peel are pre-carbonized for an hour at 150°C. The pre-carbonized orange peel is next ground and sieved to obtain self-adhesive carbon grain with a grain size of less than 100 micron. KOH at concentrations of 2 M, 3 M, and 4 M was used in the chemical activation process. The activation process lasted 22 hours and was mixed at room temperature with a magnetic stirrer. Microwave irradiation was carried out for 15 minutes with a power output of 630 Watt. After this, the activated carbon was washed many times with distilled water to eliminate any remaining organic or mineral residue, indicated by the neutral pH, and then dried for 24 hours at 105°C. OP-ACxM was the designations for the activated carbon samples, where is x as KOH concentration. X-ray diffraction (XRD), scanning electron microscopy (SEM), energy dispersive X-ray (EDX), and Fourier transform infrared (FTIR) were used to characterize the activated carbon samples.

3. Result and Discussion

As shown in Figure 1, the XRD pattern reveals that all of the OP-ACxM have a turbostratic structure, as indicated by the plane of (002) and (100) at two angles approximately 21° and 43° [4]. This shift denotes a change in the distance between atoms, which results in a shift in the distance between planes. The samples in this turbostratic model are assumed of graphite-like microcrystallites and are bordered by a cross-linking network made up of many graphite-like layers stacked roughly parallel and equidistantly, with each layer having a random orientation [4]. Bragg's equation \( n\lambda = 2d \sin \theta \) was used to get the interlayer spacings \( d_{002} \) and \( d_{100} \) [17]. The \( L_c \) and \( L_a \), were calculated using

![Figure 1. XRD patterns of OP-ACxM](image-url)
the Scherrer equation based on broadening peak (002) and (100) $L_{c,a} = \frac{k\lambda}{\beta_{c,a} \cos \theta}$ [18,19], where k is constant, $\lambda$ is X-ray wavelength, and $\beta$ is FWHM, respectively. Table 1 shows the $d_{002}$, $d_{100}$, $L_c$, $L_a$, $L_c/L_a$, and $N_p$ of OP-ACxM based on XRD data.

| Samples     | $d_{002}$ (nm) | $d_{100}$ (nm) | $L_c$ (nm) | $L_a$ (nm) | $L_c/L_a$ | $N_p$ |
|-------------|----------------|----------------|------------|------------|-----------|-------|
| OP-AC2M     | 4.141          | 2.151          | 10.505     | 4.259      | 2.466     | 2.536 |
| OP-AC3M     | 4.403          | 2.181          | 12.418     | 20.072     | 0.618     | 2.820 |
| OP-AC4M     | 4.261          | 2.095          | 19.288     | 1.804      | 10.691    | 4.574 |

With increasing KOH concentrations, the stack height ($L_c$) was increased. The higher $L_c$, the greater the surface area of activated carbon and the number of pores in activated carbon [20]. The KOH concentrations do not significantly affect the interlayer spacing of activated carbon. The microcrystallite dimension of OP-ACxM is affected by changes in KOH concentration, but the microstructure is not affected.

Figure 2 depicts the surface morphology of OP-ACxM for the OP-AC2M, OP-AC3M, and OP-AC4M samples, respectively. The surface morphology has microporous, and there are open huge macropores (in the order of microns) between grains of various sizes. The OP-AC4M appears to have more pores than the OP-AC2M and OP-AC3M, demonstrating that the microstructure of activated carbon is affected by KOH concentration. The inclusion of the KOH activator to the chemical activation procedure, together with assisted microwave irradiation, resulted in the formation of additional pores on the activated carbon [21].

Figure 2. Surface morphology of (a) OP-AC2M, (b) OP-AC3M, and (c) OP-AC4M
Carbon, oxygen, and potassium are found in the elemental compositions of OP-ACxM as determined by energy dispersive X-ray (EDX) characterisation. Chemical activation can remove impurities and gases from carbon, resulting in activated carbon that is high in carbon and low in oxygen [21]. The elemental contents of activated carbon are affected by KOH concentrations, where oxygen and non-carbon elements are evaporated, resulting in an increase in carbon content [22].

### Table 2. Elemental contents of OP-ACxM

| Samples   | Carbon (wt%) | Oxygen (wt%) | Pottasium (wt%) |
|-----------|--------------|--------------|-----------------|
| OP-AC2M   | 69.71        | 26.48        | 3.81            |
| OP-AC3M   | 50.51        | 19.12        | 2.96            |
| OP-AC4M   | 66.55        | 29.95        | 2.22            |

The goal of the Fourier transform infrared (FTIR) study is to determine which functional group each sample belongs to. The FTIR spectra of OP-ACxM with a wave number in the range of 4500-400 cm\(^{-1}\) are shown in Figure 3. All of the spectra are similar, as can be observed. At 3208 cm\(^{-1}\), there is a wide broadband, which is especially intense for the O-H stretching vibrations [23]. The C-H stretching is assigned to the band at 2886 cm\(^{-1}\). The absorption in the wavenumber domain of 1683 cm\(^{-1}\) is assumed to be C=O (carbonyl), whereas the absorption in the wavenumber domain of 1558 cm\(^{-1}\) is assumed to be C=C bond stretching vibrations in the benzene rings [24]. C-O significantly causes absorption in the wavenumber domain of 1058 cm\(^{-1}\) (secondary alcohol). The activation of KOH resulted in an increase in functional groups.

**Figure 3.** IR spectrum of OP-ACxM

### 4. Conclusion

The production of activated carbon prepared from orange peel has been successfully reviewed. The production was based on a pre-carbonization and chemical activation procedure with assisted microwave irradiation. The physical properties of activated carbon were changed by the KOH activator. The OP-ACxM has a turbostratic structure, as evidenced by the presence of (002) and (100) planes at 2\(\theta\) angles about 21° and 43° in the XRD pattern. With a Lc of 19.288 nm, the OP-AC4M sample had the greatest L\(_v\) value and the lowest L\(_a\) value of 1.804 nm. In OP-ACxM, the FTIR reveals the presence of O-H, C-H, C-C, C=O, C=C, and C-O.
Acknowledgements
The author wishes to express his gratitude to LPPM Universitas Riau for providing financial assistance through the Penelitian Bidang Ilmu grant (contract no. 662/UN.19.5.1.3/PT.01.03/2021).

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