

The Influence of Microwave Oven Output Power and Catalytic Temperature on Products Yields of Aseptic Packaging Waste Pyrolysis

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Abstract- Aseptic packaging waste (APW) is one of the waste that was made from multi-layers of materials causing difficult processing. In here double reactor microwave pyrolysis is proposed to treat the waste, the mixture of APW and absorber was placed in a microwave reactor, and the catalyst was placed in the secondary reactor made from stainless tube. Activated carbon (AC) as absorber and natural zeolite (NZ) as catalyst were used on the process. The microwave output power (300 W, 450 W, 600 W, and 800 W) and the catalytic temperature (350°C, 400°C, 450°C, and 500°C) had a significant effect in this study: the yield of gas production improved from 14% to 65% at the uncatalyzed process as the microwave output power (MOP) increased from 300 W to 800 W. When the NZ was used, the gas yield increased from 47% to 58% at 600 W as the catalytic temperature increased from uncatalyzed process to 500°C of catalytic reactor temperature. A larger amount of syngas (H₂+CO₂) was produced as MOP and catalytic temperature increased. The optimum condition to microwave pyrolysis of APW using NZ as catalyst and AC as absorber are 450°C of catalytic reactor temperature and 800 W of the MOP. The gas products from this research have a heating value of 20.82 - 23.57 MJ/m³. These findings suggest the possibility of using the gases as a fuel or feed gas for chemical synthesis. We hope that pyrolysis gas can be produced on a factory scale to help protect the environment.

Keywords Microwave pyrolysis, natural zeolite, microwave output power, catalytic temperature, aseptic packaging waste.

1. Introduction

Aseptic packaging is a package/box of drinks such as juice, milk, tea, yoghurt, etc. The increasing use of aseptic packaging causes the amount of its waste to increase accordingly. Based on data from the Directorate General of Waste Management, Hazardous Waste and Toxic Materials (Ditjen PSLB3) of the Ministry of Environment and Forestry, in 2016 there were 65 million tons of waste generated in

Indonesia. This figure is an increase of one ton compared to 2015. Meanwhile, APW is a small part of the total waste amounting to around 0.1% of the total waste heap in 2016 in Indonesia or around 63,000 tons of APW, increase 5% every year [1]. Thus, it is necessary to treat Aseptic Packaging Waste (APW) into fuel that will have economic value and be good for the environment. Pyrolysis is an alternative that can be used to treat waste, especially aseptic packaging waste [2].

Pyrolysis is one of biomass and waste's most promising thermal degradation pathways utilizing heat and chemical conversion for acquiring energy resources [3]. The process converts biomass or feedstock into solid (char), liquid, and gaseous fractions under low oxygen temperature [4]. The yields and compositions of products vary depending on the operating variables, such as feedstock, temperature, size of particle, residence time, reactor configuration, heating rate, catalyst type, catalytic temperature, and environment [5]–[12]. In recent decades, renewable fuels and biofuels have piqued attention as viable alternatives to fossil fuels [13]. Recent pyrolysis researches have optimized reaction conditions and intended products by using innovative heating technologies, such as microwave heating [14]–[16].

Pyrolysis using microwave heating or microwave pyrolysis is a process of microwave-heated thermal that takes place under low oxygen temperature. Microwave pyrolysis has some benefits over conventional heating in terms of overcoming limitations, such as uniform temperature distribution could be achieved [17]. In the microwave pyrolysis, the microwave absorber plays an important role [18]. A microwave absorber is required because raw material does not absorb microwaves well. Many researchers have studied the method by using a variety of feedstocks. Microwave pyrolysis has been employed to pyrolyze large size biomass, and this study validates the direct utilization of large-size biomass from microwave pyrolysis [19]. Microwave-induced pyrolysis of seed pits has produced gas with composition of CO (26.01 vol.%), CO₂ (11.04 vol.%), H₂ (8.54 vol.%), and CH₄ (48.46 vol.%) [20]. Microwave pyrolysis has the ability to generate H₂-rich fuel gas [21].

Catalyst can play a major role in the conversion of biomass/feedstock to pyrolysis products. The catalyst optimizes the targeted reactions, decreases reaction times and temperature, improves pyrolytic product quality, and increases the overall process efficiency [14]. Catalyst's acidity, surface area, size of pore, and volume are the major characteristics that influence the pyrolysis process [22]. The Si/Al ratio of the catalysts affected the gases product composition with the more basic catalysts producing more CO and the strong acidic catalyst producing more H₂ [23].

However, the usage of catalyst is the most expensive part of waste recycling. Reducing the cost of catalysts for small-scale applications is a challenging problem. Natural Zeolites (NZ), which could be found in many areas across the world, including Indonesia, might be utilized to replace the commercial catalysts for this purpose. Pyrolysis with NZ can reduce the liquid fraction and increased the gaseous fraction [24].

The study aims to evaluate the pyrolysis product of APW using microwave reaction system with the addition of Natural Zeolite (NZ) as catalyst. The importance of operating parameters has been the focus of a few microwave-pyrolysis studies. Studies on microwave pyrolysis at constant temperatures and the addition of catalysts in the secondary reactor have not been done. The variables of this study are MOP and catalytic reactor temperature. The alternative purpose of the present work is to study the behaviors of APW through microwave pyrolysis with/without catalyst.

Finally, the potential of direct utilization of APW through microwave catalytic pyrolysis for production of fuel is investigated according to the yields and quality of the end products.

2. Methodology

2.1. Materials

The APW used in this research is the Tetra Pak product, known as the Tetrapak package. The components in tetrapak are generally kraft paper (about 70%), low-density polyethylene (LDPE, about 25%), and aluminium foil (about 5%). They are arranged in six ordered layers from outside to inside: LDPE, paper, LDPE, aluminium foil, LDPE, LDPE. The sample was obtained from household garbage bins. The waste was cut to a uniform size of about 5 mm x 5 mm. The physical appearance of APW after cutting is shown in Fig. 1. The composition of APW is listed in Table 1 from the proximate analysis. The sample was washed before being oven-dried at 105°C for 2 h. After that, it is subjected to the pyrolysis process.



Fig. 1. The feedstocks from aseptic packaging wastes after cutting.

Table 1. Proximate analysis of aseptic packaging waste (APW)

Characteristics	wt. %
Moisture ^a	10.13
Fixed carbon ^a	3.72
Volatile matter ^a	73.47
Ash ^a	7.38
Aluminium foil ^a	5.3

^a: air dry based

Table 2. Chemical analysis (SEM-EDX) of natural zeolite (NZ)

Element	wt. %
Si	26.19
Al	8.73
O	65.08
Total	100

In this research absorber will be used in the form of Activated Carbon (AC). The carbon used is coconut shell charcoal obtained from a charcoal industry in Yogyakarta, Indonesia. Before microwave pyrolysis analysis, the AC was ground by a rotary speed mill (P-14, Fritsch) and then sieved to collect samples in 40-80 mesh size. After that, the AC was oven-dried at 105°C for 2 h.

NZ was collected from Klaten District, Central Java, Indonesia. In each experiment, 50 g of NZ were used. Before use, the catalyst was crushed to obtain particles measuring 40-80 mesh. Then, the catalyst was washed and heated at 105°C for 2 h. The composition is listed in Table 2 from the SEM-EDX analysis.

2.2. Experimental apparatus and procedures

The microwave pyrolysis experiments were conducted in the Energy Conversion Laboratory, Universitas Gadjah

Mada, Yogyakarta, Indonesia. An Electrolux microwave oven EMM2308X model with frequency of 2.45 GHz and 1300 W electrical input power used in this study. A 50 g of APW was mixed with 25 g of carbon. Afterwards, the mixture was placed in a reactor made from glass (1000 ml volume).

The pyrolysis was carried out in nitrogen atmosphere using four variations of MOP 300 W, 450 W, 600 W, and 800 W, for 30 minutes. The experiments were carried out from room temperature until they reached 450°C in the microwave reactor.

The thermal and catalytic decomposition of volatile matter took place in the secondary reactor. A stainless-steel tube with a diameter of 25 cm filled with catalyst particles is placed into an electrically heated tube furnace. The schematic system diagram is displayed in Fig. 2 for pyrolysis with single reactor and Fig. 3 for double reactor catalytic pyrolysis.

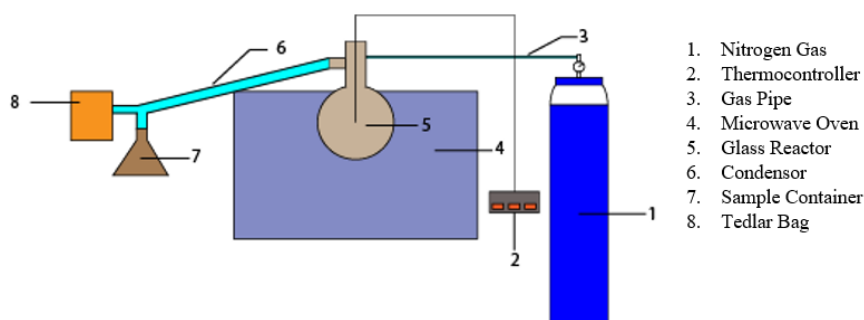


Fig. 2. The schematic diagram of the experimental apparatus with single reactor

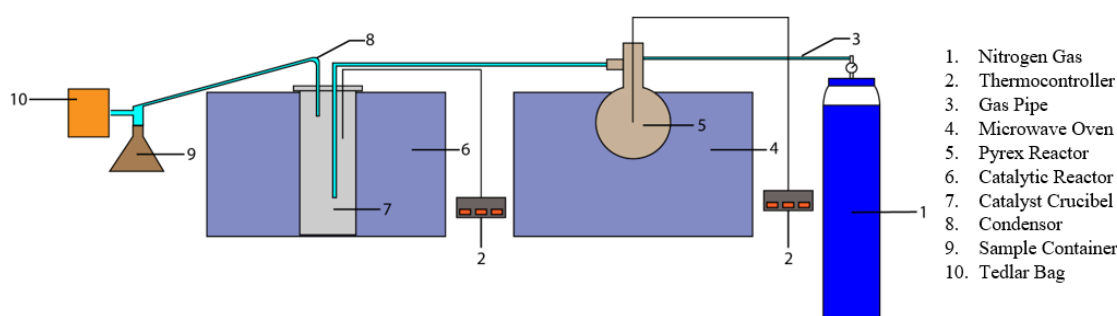


Fig. 3. The schematic diagram of the experimental apparatus with double reactor catalytic pyrolysis

Nitrogen gas in this process was used as flushing gas. Before the pyrolysis process began, nitrogen gas was flowed to the primary reactor to remove air. The flushing gas was vented through the apparatus at a flow rate of 100 ml/minutes. The primary reactor used a microwave oven so that the heat supply was generated from microwaves. The sample was decomposed into volatile matter. The volatile matter flowed into the condenser. Char products and aluminium foil were left in the glass reactor. The condensed product, namely liquid and wax, was collected in a container made from Pyrex glass. Meanwhile, gas products were collected in Tedlar bags (500 ml volume, TECHINSTRO).

For microwave pyrolysis of APW with NZ as absorber, four variations of catalytic temperature were used (350°C, 400°C, 450°C, and 500°C).

A gas syringe was used to collect gas samples, and then analysed using a Hewlett-Packard model 5989AMS Gas Chromatography-Mass Spectrometry (GC-MS) with ionization energy of 70 eV in the electron potential source of error is that the temperature profile observed by an optical pyrometer is only corresponding to the surface temperature of the sample.

Proximate analysis was conducted using a TGA-501 by Leco to determine the content of fixed carbon, volatile matter, and ash of APW. The samples were heated from room temperature to 400°C at a heating rate of 25°C/min with a final holding time of 20 min, and the N₂ flow rate of 0.1 l/min.

2.3. Determine the pyrolysis yields, lower heating value (LHV), and total energy

The yield of each product was calculated from Equation (1)-(5) [23]:

$$\text{Aq. phase yield (wt. \%)} = \frac{\text{mass of aq. phase produced}}{\text{mass of APW}} \times 100\% \quad (1)$$

$$\text{Wax yield (wt. \%)} = \frac{\text{mass of wax produced}}{\text{mass of APW}} \times 100\% \quad (2)$$

$$\text{Char yield (wt. \%)} = \frac{\text{mass of char produced}}{\text{mass of APW}} \times 100\% \quad (3)$$

$$\text{Al. foil yield (wt. \%)} = \frac{\text{mass of al. foil produced}}{\text{mass of APW}} \times 100\% \quad (4)$$

$$\text{Gas yield (wt. \%)} = 100\% - \text{Aq. phase yield} - \text{Wax yield} - \text{Char yield} - \text{Al. Foil Yield} \quad (5)$$

The heating value of gas yields can be calculated by the following formula Dai *et al.*, [25]:

$$\text{LHV} = [(30.0 \times \text{CO}) + (25.7 \times \text{H}_2) + (85.4 \times \text{CH}_4) + (151.3 \times \text{HC}_x) \times 4.2] \text{ (kJ/m}^3\text{)} \quad (6)$$

CO, H₂, CH₄, and HC_x are the volume percentage of CO, H₂, CH₄, and the other light hydrocarbons (C₂₊) in the total of gas products, respectively.

The total energy (kJ) of the gas yields was calculated from Equation (7):

$$\text{Total energy (kJ)} = \text{LHV} \times \text{volume the gas products} \quad (7)$$

volume gas is calculated from mass of gas, its from mass balance using ideal gas law.

3. Results and Discussions

3.1. The influence of microwave output power on pyrolysis products yield

In this instance, the product of APW pyrolysis contained wax and aqueous phase. The solid from the main reactor consisted of ash, aluminium foil, and microwave absorber. Aluminium foil and ash can be separated from the microwave absorber by sieving. The aluminium foil is produced without any structural or chemical modifications. The microwave pyrolysis product from APW is classified into five groups: aqueous phase, wax, char, gas products, and aluminium foil.

The liquid product in this research consists of an organic aqueous phase (formed from the degradation of cellulose) and a viscous phase (wax). When cellulose and lignocellulosic material degraded, a large number of water-

soluble or insoluble oxygenated compounds were generated. Wax is a constitutive product of the pyrolysis of polyolefins (polypropylene, low-density polyethylene/LDPE, and high-density polyethylene/HDPE) [26].

Fig. 4 showed the pyrolysis products of APW using a microwave oven with coconut shell carbon under different MOP (300 W, 450 W, 600 W, and 800 W) and four catalytic temperature variations (350°C, 400°C, 450°C, and 500°C). The difference in the output power could result in the difference of heating rate and the time to reach the final temperature. Lower output power could slow down the cracking reaction so that it took a longer processing time. In this study, the pyrolysis process was carried out at the same duration of 30 min, so that the amount of pyrolysis products was varied. At MOP below 300 W, the final temperature that could be achieved was 210°C and no pyrolysis of the samples could be observed at this power levels [27].

As seen from Fig. 4, in all of the catalytic temperatures, the higher the MOP, the lower the yield of char product. This could be because as microwave power increased, the microwave density of the cavity and the microwave absorption capacity of APW increased. As a result, the overall temperature of APW increased, making it more beneficial to decompose longer-chain molecules into shorter-chain molecules. Therefore, the yield of char residue decreased while the liquid and gas product increased. So, when the MOP increased from 300 W to 800 W at the 350°C catalytic temperature the char residue decreased from 69 % to 25 %, aqueous phase decreased from 4 % to 0 %, wax product decreased from 6 % to 1 % and the amount of the gaseous product increased from 16 % to 69 %. At the catalytic temperature of 400°C the char residue decreased from 69 % to 23 %, aqueous phase decreased from 4 % to 0 %, wax product decreased from 4 % to 1 % and the amount of the gaseous product increased from 18 % to 71 %. At the catalytic temperature of 450°C the char residue decreased from 66 % to 18 %, aqueous phase decreased from 5 % to 4 %, wax product decreased from 3 % to 0 % and the amount of the gaseous product increased from 21 % to 73 %. Finally, at the catalytic temperature of 500°C the char residue decreased from 64 % to 23 %, aqueous phase decreased from 6 % to 5 %, wax product decreased from 2 % to 0 % and the amount of the gaseous product increased from 23 % to 67 %. This finding has a similar correlation with another study [28]. The possibility is that the increasing MOP easily produced smaller molecules during secondary reaction of liquid product. The products that resulted from the microwave pyrolysis process were due to the thermal breakdown in the first reactor and catalytic degradation of volatiles in the second reactor. In secondary reactions, the main products may continue to depolymerize through the cracking processes resulting gas products, or cross-link through condensation reactions, resulting in wax formation.

The above analysis indicated that 800 W was the best MOP under different catalytic temperatures to result in the highest gas yield of 73 % with the lowest wax production.

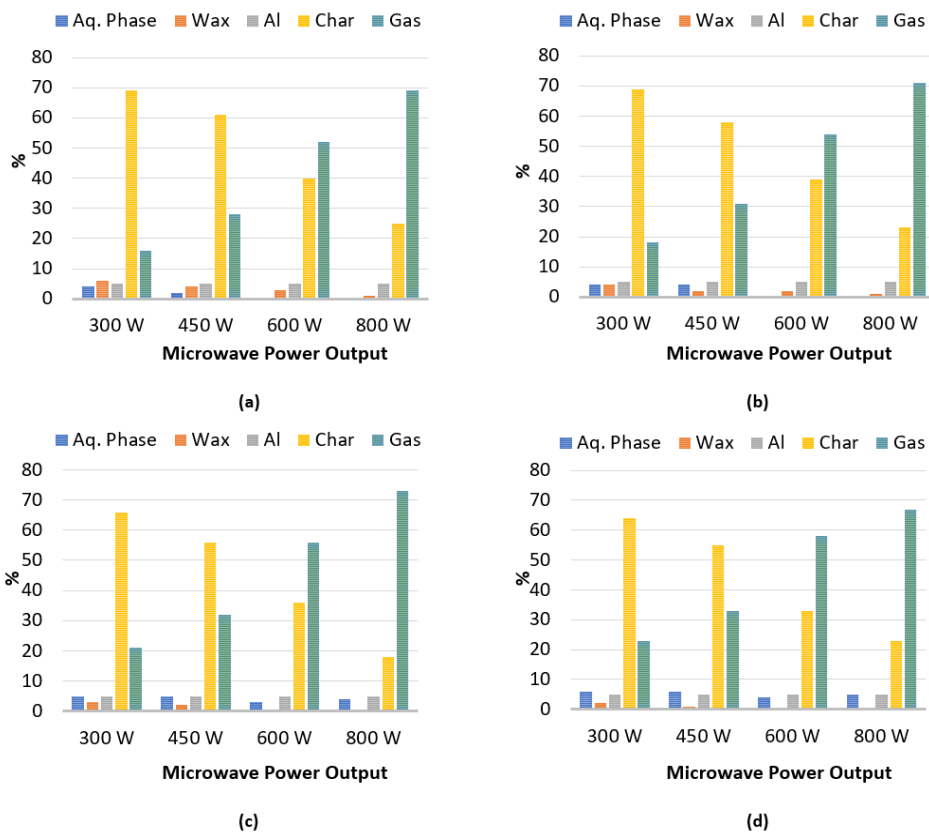


Fig. 4. Pyrolysis products yield using activated carbon as absorber under different microwave output power with catalytic temperature (a) 350°C, (b) 400°C, (c) 450°C, and (d) 500°C

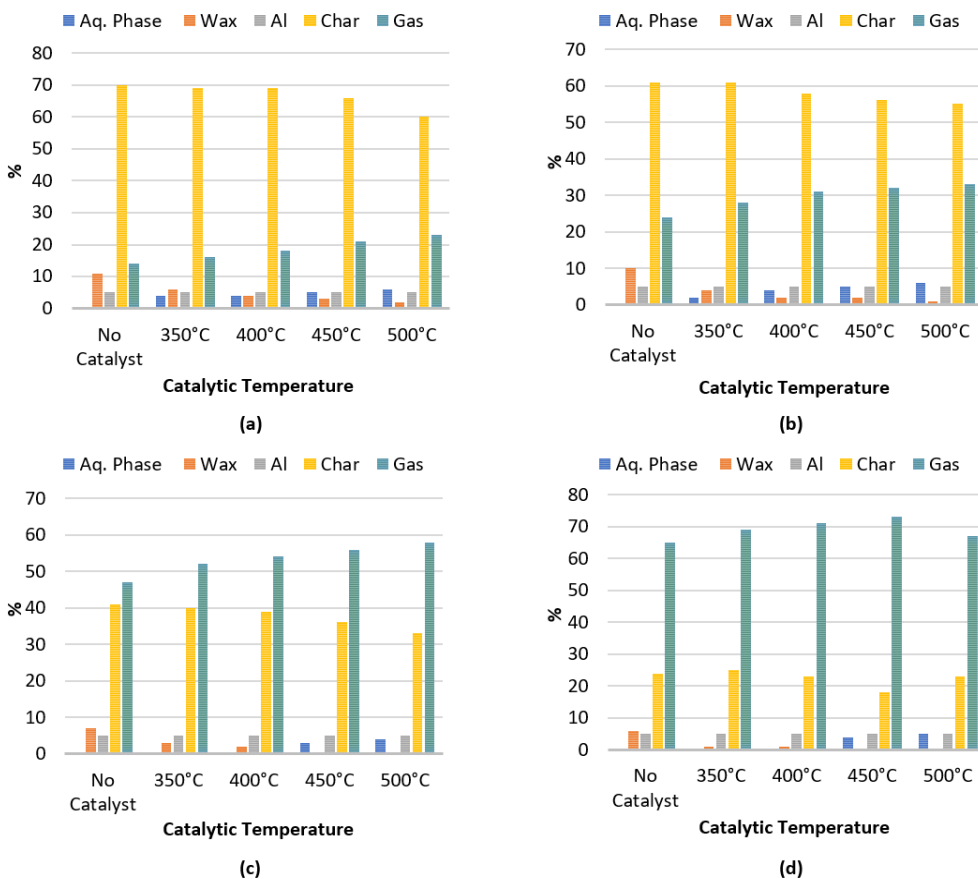


Fig. 5. Pyrolysis products yield using activated carbon as absorber under different catalytic temperature with microwave output power (a) 300 W, (b) 450 W, (c) 600 W, and (d) 800 W

3.2. The influence of catalytic temperature on pyrolysis products yield

Fig. 5 depicted the effect of NZ catalytic temperature on the microwave pyrolysis product yields at 300 W, 450 W, 600 W, and 800 W for 30 min. For all the tests, an increase in catalytic temperature led to an increase in gas yields while liquid yields and char residue decreased. When the NZ was used, the yield of aqueous phase and gas was higher, and the yield of wax and char was lower than the process without catalyst. This result is similar to Jerzak *et al* [26], which suggested using catalyst to reduce wax formation. For example, in 600 W there was an initial decrease in wax yield from 7% in the absence of catalyst to 3% wax yield followed by a further decrease to 0% as the catalyst temperature was increased from 350°C to 500°C. In addition, the gas yield increased from an uncatalyzed gas yield of 47 – 52% at 350°C catalytic temperature to 58% at 500°C.

It indicated that the catalytic temperature had a higher influence on gas production than the presence of NZ catalyst. The increase in gas yield with an increase of catalytic temperature had been reported in the literature [23]. Catalyst increased the cracking process rate, resulting in a higher yield of gases but lowering the yield of liquid oil [29]. The catalyst like NZ with low BET surface area, microporous structure, and low acidity showed good catalytic performance for the degradation of raw material (*i.e.* the mixture of plastic) that may increase the production of gases [30], as observed in this study.

Fig. 5 showed that if the catalytic temperature got higher, the yield of gas increases. However, something different happened at 800 W MOP, where the gas product decreases after a catalytic temperature of 450°C from 73% to 67%. Therefore, the optimum condition to microwave pyrolysis of APW using NZ as catalyst and AC as absorber were at 450°C catalytic temperature and 800 W MOP.

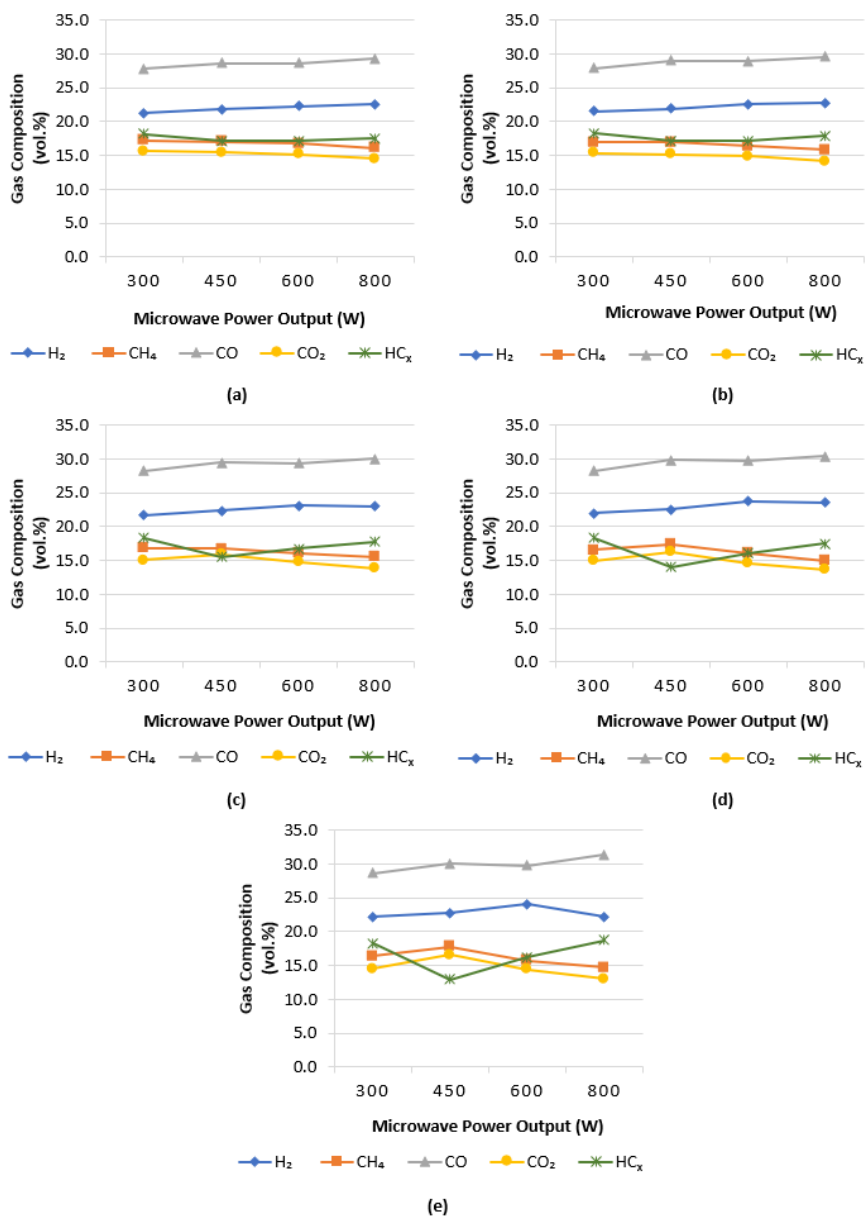


Fig. 6. Composition of gas under different microwave output power with catalytic temperature (a) without catalyst, (b) 350°C, (c) 400°C, (d) 450°C, and (e) 500°C

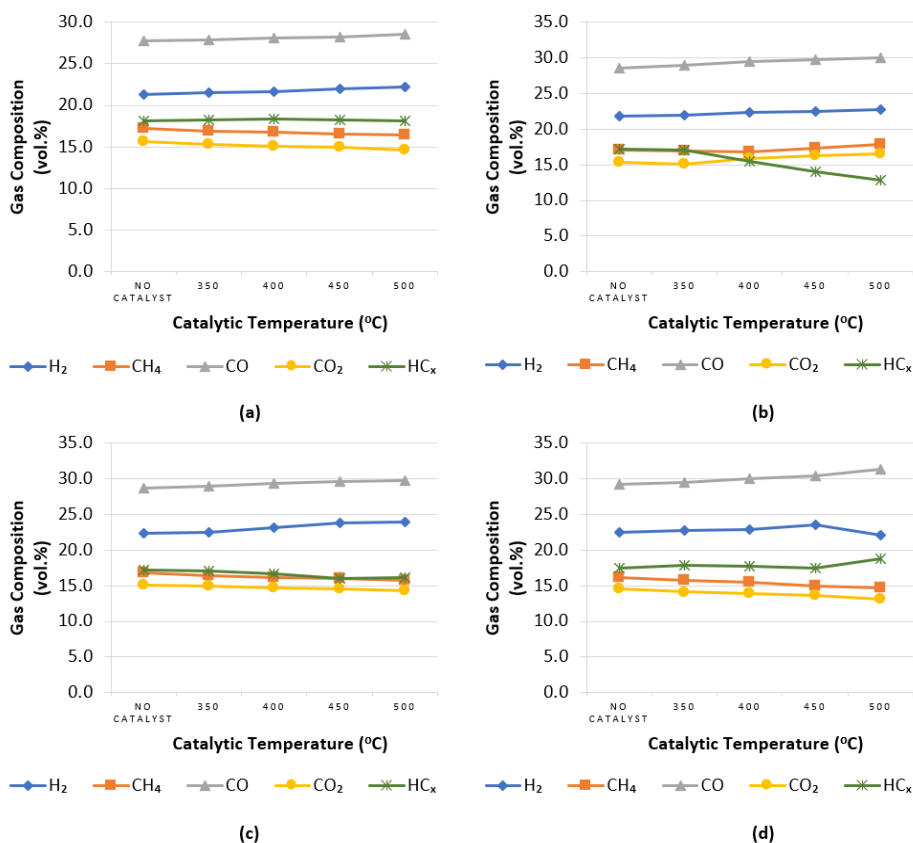


Fig. 7. Composition of gas under different microwave output power (a) 300 W, (b) 450 W, (c) 600 W, and (d) 800 W

3.3. Gas composition

The gas products were generated as a result of tar cracking, wax cracking, char breakdown at high temperatures, and interactions between the species formed during pyrolysis. The composition of gas products from microwave pyrolysis of APW at different MOPs is shown in Fig. 6. Analyses of the gaseous products composition have shown H₂, CH₄, CO, CO₂, and light hydrocarbons. When the MOP was 300 W, the primary composition of the gas products was H₂ (21.30 vol.%), CH₄ (17.20 vol.%), CO (27.80 vol.%), CO₂ (15.60 vol.%), and light hydrocarbons (18.10 vol.%). With the increase in MOP, the content of H₂ increased from 21.30 vol.% (300 W) to 23.55 vol.% (800 W) and CO₂ content decreased from 15.60 vol.% (300 W) to 13.10 vol.% (800 W). The yield of syngas (CO + H₂) exceeded 45 vol.% in the total of gas products, and this indicates that microwave pyrolysis of APW had an advantage in producing syngas. Dehydrogenation processes in char and oil, such as aromatization, condensation, and alkene formation, generated hydrogen. The significant degree of deoxygenation produced during the pyrolysis experiment is responsible for the high CO₂ and CO contents in the pyrolysis gas products. The content of CO increased slowly with the increase in MOP, accounting for 27.80-31.35 vol.% of the total of gas products. The CO₂ yield decreases when the MOP is increased, which appears to contribute to the higher production of CO₂. With increasing MOP, the content of CH₄ decreased, implying that high MOP promotes CH₄ breakdown and dry reforming processes. In addition, CH₄ is

thought to form mainly due to the splitting of C-O bonds during decomposition of lignin.

Fig. 7 depicted the composition of gas products from microwave pyrolysis of APW at different catalytic temperatures. Same as composition of gas under different microwave output powers, the gas analyser enabled the measurement of H₂, CO, CH₄, CO₂, and light hydrocarbons (HC_x). The catalytic temperature has the greatest impact on the composition of the H₂ volume percentage, which increased 22.30 vol.% (no catalyst) to 24.00 vol.% (500°C) at 600 W of MOP. Another significant of syngas, CO, increased from 28.65 vol.% in the absence of catalyst process to 29.75 vol.% (500°C) at 600 W. The light hydrocarbons (HC_x) were detected in small but not negligible amounts in the gas products, because these compounds have significantly greater heating values than other gas components, therefore they are particularly important for the ultimate use of these gases as fuels.

It was found in the study, that the optimal operating condition to obtain a high concentration of the yield of syngas (CO + H₂) was at 500°C of catalytic temperature using NZ as catalyst and 600 W of MOP using AC as absorber. The heating value of the gas products from microwave pyrolysis of APW using AC as absorber and NZ as catalyst was 20.82 - 23.57 MJ/m³. These results suggest that the gases might be utilized as a fuel or as a feed gas for chemical synthesis [31].

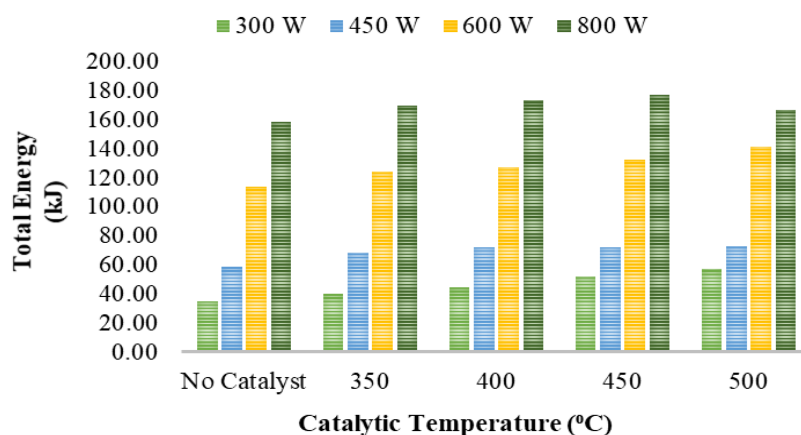


Fig. 8. Total energy of gas yield from microwave pyrolysis of APW

The effects of MOP and catalytic reactor on the total energy of the gas products was showed in Fig. 8. With increasing MOP from 300 W to 800 W the total energy was increased from 34.85 kJ to 158.62 kJ at uncatalyzed process, from 39.90 kJ to 169.62 kJ at 350°C of catalytic temperature, from 44.99 kJ to 173.74 kJ at 400°C of catalytic temperature, from 52.28 kJ to 176.80 kJ at 450°C of catalytic temperature, and from 57.21 kJ to 167.03 kJ at 500°C of catalytic temperature.

4. Conclusions

The effect of MOP and catalytic temperature on microwave pyrolysis using AC as absorber and NZ as catalyst has been studied. The experiments were conducted using MOP at 300 W to 800 W and catalytic temperature at 350°C to 500°C aiming to determine the quantity of pyrolysis products and the quality of the gas products. The result showed that this experiment's yield product consists of aqueous phase, wax, char, gas, and aluminium foil. When the MOP increased at the uncatalyzed process, the char, aqueous phase, and wax product decreased from 70 % to 23 %, 4 % to 0 %, and 11 % to 0 %, respectively. However, the gaseous yield increased from 14 % to 73 % when the microwave output power increased. When the NZ was used at the MOP of 600 W, the gas yield increased from an uncatalyzed process of 47 – 52 % at 350°C catalyst temperature to 58 % at 500°C. In this process a large amount of H₂, CH₄, CO, CO₂, and other light hydrocarbons were produced. The yield of syngas (CO + H₂) exceeded 45 vol.% in the total of gas products, and this indicated that microwave pyrolysis of APW had an advantage in producing syngas. The heating value of the gas products was 20.82 - 23.57 MJ/m³, which indicated that they might be utilized as a fuel or as a feed gas for chemical synthesis.

The behaviour of APW samples was studied in this article, and it was discovered that such a method is a promising strategy for APW feedstock recycling. Variations in the process, such as the MOP and the catalytic temperature, allow to produce high-quality gases and chemicals from low value APW.

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