Effect of Activated Carbon and Catalyst on Physical and Chemical Properties of Pyrolytic Oil from Microwave Assisted Pyrolysis of Automotive Paint Sludge (APS)

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Keywords: microwave pyrolysis, activated carbon, automotive paint sludge, catalytic microwave pyrolysis, catalyst ZSM-5.

Abstract.

Automotive paint sludge (APS) contains valuable material which can be recovered by microwave-assisted pyrolysis as studied by previous researchers. In this study, combined application of microwave absorber and catalyst are studied in order to study the effect of both components in microwave-assisted pyrolysis of APS. Activated carbon and ZSM-5 acted as microwave absorber and catalyst, respectively were used to investigate their effect on pyrolytic oil produced from APS. By using One-Factor-At-Time (OFAT) analysis, results showed that at 1000W power level, 30 minutes radiation time, 6% activated carbon loading and 1.5% catalyst loading, the process was capable to produce highest oil yield of 7.75% pyrolytic oil as compared to other parameters. On the other hand, physical and chemical properties of pyrolytic oil produced via this process was also improved.

Introduction

Automotive paint sludge (APS) is mainly generated from painting process in car manufacturing plant. Car manufacturing process consists of four main stages which are stamping, welding, painting, and assembly. During the painting process in the spray booth, excess paint was accumulated in the water stream in the tank under the spray booth. The excess spray paint were injected with paint killer and then coagulates to form APS and collected at the sludge tank [1].

Based on data, demands of car are increasing steadily over the year which can be seen in Fig. 1, in which contributing to the production of APS [2]. There are in average 200 tonne of APS generated each year in Malaysia and the primary sources of hazardous waste in automotive manufacturing plant is mainly generated from automotive painting process, which comprises of 80% of environmental concerns in manufacturing plants [3], [4]. Table 1 showed chemical composition of APS. Xylene, a cyclic hydrocarbon, is the highest percentage of compound in APS. Short term exposure to xylene caused irritation to sensory organ while long term exposure lead to respiratory problem. According to Zorpas and Inglekazis (2012), painting and coating account for the largest share of environmental impacts (62%) during the manufacturing of automobiles [5].

Treatment of APS before being disposed of at the landfill is quite tedious and time consuming. Hence, there is a need to search for alternative way of disposal of APS that requires short period of treatment at the same time is able to recover valuable products as shown in Table 2.
Fig. 1: Production of vehicles in Malaysia from 1980 to 2017 [2]

TABLE 1: AUTOMOTIVE PAINT SLUDGE CHEMICAL COMPOSITION [6]

| Raw material composition | wt.% |
|--------------------------|------|
| Xylene                   | 43.00|
| Solvesso 150             | 19.80|
| Toluene                  | 15.00|
| Ethyl Acetate            | 5.75 |
| Ester                    | 4.88 |
| Glycol Ether             | 2.69 |
| Acrylic Resin            | 1.54 |
| Cellosolve Acetate       | 1.46 |
| Amino Resin              | 1.12 |
| Acrylic copolymer        | 1.12 |

TABLE 2: ELEMENTAL RESULTS OF APS FROM X-RAY FLUORESCENCE SPECTROSCOPY (XRF) TEST [7], [8]

| Element                   | Concentration |
|---------------------------|----------------|
| Aluminium oxide, Al₂O₃    | 18.01 wt%      |
| Silicon dioxide, SiO₂     | 2.15 wt%       |
| Titanium dioxide, TiO₂    | 54.21 wt%      |
| Barium oxide, BaO         | 11.26 wt%      |
| Bromine, Br               | 300 ppm        |

Microwave-assisted pyrolysis is a process in which decomposition of hydrocarbon materials by heating them in the absence of oxygen or any other reagents using microwave radiation energy [9]. Products of the process are pyrolytic oil, char and syn gas. Microwave-assisted pyrolysis has many advantages as compared to conventional pyrolysis including uniform, rapid, and fast heating without pre-treatment process such as drying and grinding [10],[11].

Microwave-assisted pyrolysis application towards energy and valuable material recovery is vast. Various studies have been conducted to recover oil from waste material such as lignocellulosic biomass (wood, rice husk and rice straw) by Bo Zhang et al. [10],[12], Lam et al. [13] on waste palm...
oil, and J.H Ng et al. [14] used crude glycerol of waste palm oil to produce bio-oil. Other non-bio material such as oil waste from oil shipping and coal solid can also be pyrolyzed with microwave-assisted pyrolysis to recover oil from the material which has been reported by Wan Adibah et al. [15] and Faisal Mustaq et al. [16].

In this study, the effect of activated carbon and catalyst ZSM-5 were investigated towards pyrolytic oil yield with One-Factor-At-Time (OFAT) analysis.

Materials and Method

A. Materials

Automotive paint sludge (APS) which is the main feedstock of this study was collected from an automotive manufacturer in Klang Valley, Malaysia. APS is in slurry form was collected from the water wash chamber under spray booth section. No pre-treatment of APS is needed prior to microwave assisted pyrolysis. Weight of the sample is fixed at 200 g which yielded the highest oil product in previous study [3] and also was due to depth of penetration [17]. Meanwhile, microwave absorber (MWA) used in this study is activated carbon and catalyst used is ZSM-5.

B. Methods

Domestic microwave (2.45 GHz) had been used to pyrolyze APS. To investigate the effect of activated carbon and catalyst ZSM-5 on pyrolytic oil, APS was placed in a quartz reactor with fixed loading amount of 200 g at 1000 W, 30 min and fixed amount of catalyst of 1.5 wt.%. In this study, experiments were run based on parameter setting as shown in Table 3, Table 4, Table 5 and Table 6.

| Power (W) | Time (min) | MWA % | Catalyst % |
|-----------|------------|--------|-------------|
| 300       | 30         | 6      | 1.5         |
| 600       | 30         | 6      | 1.5         |
| 700       | 30         | 6      | 1.5         |
| 1000      | 30         | 6      | 1.5         |

| Power (W) | Time (min) | MWA % | Catalyst % |
|-----------|------------|--------|-------------|
| 1000      | 20         | 6      | 1.5         |
| 1000      | 30         | 6      | 1.5         |
| 1000      | 40         | 6      | 1.5         |
| 1000      | 50         | 6      | 1.5         |
TABLE 5: EFFECT OF ACTIVATED CARBON LOADING (MWA) PARAMETER

| Power (W) | Time (min) | MWA % | Catalyst % |
|-----------|------------|-------|------------|
| 1000      | 30         | 4     | 1.5        |
| 1000      | 30         | 6     | 1.5        |
| 1000      | 30         | 8     | 1.5        |
| 1000      | 30         | 10    | 1.5        |

TABLE 6: EFFECT OF CATALYST LOADING PARAMETER

| Power (W) | Time (min) | MWA % | Catalyst % |
|-----------|------------|-------|------------|
| 1000      | 30         | 6     | 0.5        |
| 1000      | 30         | 6     | 1.0        |
| 1000      | 30         | 6     | 1.5        |
| 1000      | 30         | 6     | 2.0        |

APS was placed in a quartz reactor and mixed thoroughly with activated carbon (microwave absorber). Meanwhile, catalyst ZSM-5 was placed in a silica crucible without a lid. The silica crucible was then placed inside the quartz reactor in the microwave. 250 ml/min of nitrogen gas was purged into microwave system for 10 minutes prior to experiment commencement in order to ensure inert condition in the quartz reactor. After experiment starts, nitrogen gas inlet was set to 150 ml/min until experiment ends. Experiment was repeated as per setting in Table 3, Table 4, Table 5 and Table 6. Nitrogen gas was used as carrier gas for inert condition preparation in the quartz reactor in many studies such as used by Lam et al. [13],[18],[19].

Three condensers were arranged in series to collect the condensable gas, whereby the oil product were collected. The arrangement of microwave-assisted pyrolysis of APS is shown in Fig.2.

Fig. 2. Arrangement of microwave pyrolysis of APS [11]
The resulting liquid product was then separated by using liquid-liquid extraction method. Hexane was used as a solvent for the extraction as it is suitable for non-polar hydrocarbon separation [20]. Hundred mililiter of hexane was added to the liquid product obtained from condenser in each cycle of experiment. The resulting liquid product from the extraction process formed an oil fraction and an aqueous fraction. The amount of oil product which has been obtained was then collected and recorded for further analysis.

Results and Discussion

A. Production of Pyrolytic Oil Using One-Factor-At-Time (OFAT)

In this section, the capability of automotive paint sludge (APS) to be converted into pyrolytic oil is studied by using One-Factor-At-Time (OFAT). As a starter, important parameters such as power, radiation time, activated carbon (AC) amount and catalyst ZSM-5 amount have been chosen in this research as these parameters impacted significantly to the production of pyrolytic oil [17], [21]–[24]. As for sample weight loading, it was mentioned by Januri (2017) that 200g of APS was fixed due to depth of penetration for microwave pyrolysis of APS due to high penetration of wavelength which resulted complete pyrolysis as compared to other sample weight loading [17]. Furthermore, in a study reported by Januri et. al (2015), APS sample weight loading of 200g produced highest oil yield from microwave assisted pyrolysis [3].

In One-Factor-At-Time (OFAT) analysis, each parameter was varied while other parameters were fixed. Subsequently, the best parameters were concluded from four sets of experiments with single determination method as described in Table 3, Table 4, Table 5 and Table 6.

Fig. 4 shows 1000W power level produced highest oil yield at a value of 7.75%, even though in Fig. 3, which it produced slightly lower liquid oil compared to other power level of 700W. It this study, liquid oil is defined as the combination of aqueous, which was the light-yellowish at bottom, and pyrolytic oil, which was the upper layer of the liquid oil.

Fig. 3. OFAT result for Effect of Power Level – Product Distribution
As to separate the layers, liquid-liquid extraction method was performed by adding hexane and eventually desired pyrolytic oil was separated from aqueous layer. The highest oil yield from power level of 1000W can be explained as the higher power level exposed to APS, the higher possibility of APS would breakdown into smaller compounds. It is due to high vibration introduced by microwave wavelength penetration and distribution onto APS, which subsequently maximized collision of molecules. Therefore, it enhanced hydrocarbon chain breakdown to low hydrocarbon compound. With regards to power level in microwave assisted pyrolysis, researchers such as Zhou et al. (2013) and Song et al. (2011) have studied the effect of microwave power level on the oil yield and agreed that higher power level produced higher oil yield in their researches [23], [25].

On the other hand, the effect of radiation time was experimented in OFAT analysis as shown in Fig. 5 and Fig. 6.
The correlation between radiation time and oil yield in OFAT analysis can be described as the increased radiation time would increase the oil yield. It can be seen from Fig. 6 that 30 minutes of radiation time resulted in the highest oil yield compared to other radiation time at the value of 7.75%. As radiation time increased to 40 minutes and 50 minutes, there were drop in oil yield observed with 6.35% and 5.65% oil yield, respectively. It can be explained at certain point, longer radiation time would increase excess reaction, which then caused high formation of syn-gas compared to pyrolytic oil. This would increase the formation of polycyclic aromatic compound (PAH) [26], which occurred due to high reaction time caused by rigorous cracking at high temperature [27].

On the other hand, effect of microwave absorber, which is activated carbon (AC) amount was observed in OFAT analysis. Activated carbon amount was denoted as microwave absorber (MWA) and depicted in Fig. 7 and Fig. 8.
From Fig. 7 and Fig. 8, it can be seen that at MWA of 6%, liquid product and oil yield were recorded as the highest with 25.68% and 7.75% respectively. Amount of activated carbon in microwave assisted pyrolysis of APS was strongly related with temperature within the reactor system during the process took place. Activated carbon was considered as a good microwave energy receptor as mentioned by Rahman et. al (2015), in which APS itself was a poor microwave energy receptor [11]. Furthermore, another study conducted by Rahman et. al (2014) and Rahman et. al (2018) noted that addition of activated carbon into APS during microwave assisted pyrolysis helped to increase amount of dielectric constant by promoting and enhancing the pyrolysis process with assistance of microwave radiation [21],[28].

Another explanation of decreasing trend of liquid product and oil yield observed with increasing amount of activated carbon was that the secondary reaction had occurred, in which process would produce more non-condensable gas compared to pyrolytic oil due to thermal cracking at a certain point of temperature as shown in Fig. 8. This could be further explained by a study conducted by Ariff et. al (2018) which described the relation between the usage of activated carbon with temperature during microwave assisted pyrolysis of APS [29].

Another parameter which was tested in OFAT analysis was effect of catalyst amount as shown in Fig 9 and Fig. 10. In this study, catalyst amount was varied at 0.5%, 1.0%, 1.5% and 2.0%. From Fig. 9 and Fig. 10, liquid product and oil yield increased as catalyst amount increased, in which liquid product and oil yield were the highest at 1.5% of catalyst amount with the value of 25.68% and 7.75%. However, the trend of liquid product and oil yield decreased as catalyst amount was increased. It can be explained that increase of catalyst amount would increase the selectivity of pyrolytic oil during the microwave assisted pyrolysis took place, as reported by Ariff et. al [29]. However, selectivity of pyrolytic oil shifted to produce more non-condensable gas as more catalyst amount was added which caused further cracking of hydrocarbon chain [30].
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![Fig. 9. OFAT result for Catalyst Loading – Product Distribution](image1)

![Fig. 10. OFAT result for Catalyst Loading – Oil Yield](image2)
B. Physical Properties of Pyrolytic Oil

Physical properties of pyrolytic oil were shown in Table 7 and it was observed that the density of catalytic oil with activated carbon (AC) and catalyst ZSM-5 was reduced as compared to previous study on pyrolytic oil without activated carbon and catalyst.

| Properties                                      | Density, kg/m³ | Viscosity, cP | Gross Calorific Value, MJ/kg | Reference          |
|-------------------------------------------------|----------------|---------------|------------------------------|--------------------|
| Catalytic pyrolytic oil (with activated carbon) | 832            | 4.62          | 39.4                         | This study         |
| Pyrolytic oil (without activated carbon and catalyst) | 920            | 12.6          | 37.8                         | [17]               |
| Gasoline                                        | 767            | 0.7           | 46.1                         | [22]               |
| Diesel                                          | 810            | 1.6 – 5.8     | 42.21                        | [31], [25]         |
| ASTM D7544-12                                   | 1100 – 1300    | 1.25 – 1.19   | 15 – 45.4                    | [32], [33]         |
| Waste Plastic                                   | 750            | 119           | 41.1                         | [34]               |
| Waste Plastic + y-Zeolite                        | 820            | 22            | 41.0                         | [35]               |
| Sewage sludge                                   | N.R            |               |                              |                    |
| Commercialized liquid fuel                      | 720 – 850      | 2.22 – 6.24   | 36 – 48                      | [36], [22], [37]   |

As per result shown in Table 7, it was observed that heavy hydrocarbon in the pyrolytic oil was broken down into low hydrocarbon compound. As compared to the standard density of pyrolytic oil in ASTM D7544-14, catalytic pyrolytic oil, density obtained was near to the commercial fuel density at 832 kg/m³. Low amount of catalyst and activated carbon loading ratios are suitable to be used in order to obtain suitable density of pyrolytic oil. It was also observed that density of catalytic pyrolytic oil in this study is lower as compared to density of previous study, which was 832 kg/ m³. Generally, presence of activated carbon and catalyst in microwave pyrolysis of APS has improved density of pyrolytic oil. Evidently, it has significantly reduced the value of density of pyrolytic oil obtained in this study when activated carbon and catalyst ratio were utilized at low range as catalyst has functioned to break down heavy hydrocarbon to low hydrocarbon compound as figured in GCMS analysis in the next section.

In addition, it was shown that calorific value of catalytic pyrolytic oil in this study was in the range of commercialized fuel calorific value of 36 – 48 MJ/kg. Nevertheless, it was slightly higher than previous study, which was 37.8 MJ/kg [17]. The result of calorific value was due to formation of more oxygenated compound found in catalytic pyrolytic oil in this study. More formation of oxygenated compound in pyrolytic oil would result in lower calorific value of fuel.
In addition, the use of catalyst ZSM-5 and activated carbon in microwave assisted pyrolysis has enhanced the physical properties of pyrolytic oil. In fact, this study has demonstrated that the use of low amount of catalyst and activated carbon has produced good properties of pyrolytic oil. Next, further investigation on chemical properties of catalytic pyrolytic oil with activated carbon has been done.

C. Chemical Properties of Pyrolytic Oil

Details on hydrocarbon compound would be elaborated with GCMS analysis for catalytic pyrolytic oil. On the other hand, comparison with GCMS result of previous study would also be explained as to compare effect of activated carbon and catalyst ZSM-5 towards chemical properties of pyrolytic oil. Ideally, it would be desirable to obtain high monoaromatics and aliphatic compounds; and low oxygenated, nitrogenated and polycyclic aromatic hydrocarbon (PAH) compounds as to enhance the physical properties, such as calorific value (CV), density and viscosity of the pyrolytic oil. The effect of high percentage of oxygenated compound includes reduction of fuel properties of pyrolytic oil, which could cause corrosion issue in combustion engine.

![Fig. 9. Chemical Properties of Pyrolytic Oil](image)

From Fig. 9, it was shown that oxygenated, nitrogenated and PAH compound has been reduced when by adding activated carbon and catalyst ZSM-5 to the microwave assisted pyrolysis process of automotive paint sludge. High percentage of these compounds are not desirable in pyrolytic oil.

High oxygenated compound, for instance, would lead to increase acidity of pyrolytic oil and reduce calorific value (CV) of pyrolytic oil. During thermal cracking process of oxygenated hydrocarbon, the first stage of reaction during the thermal cracking of oxygen contained aliphatic via dehydration and decarboxylation reaction [38]. Then, similar reaction took place in intermediate reaction during formation of aromatic compound from cyclocompound.

In addition, high percentage of nitrogenated compound would affect viscosity of pyrolytic oil and contribute to clogging formation in combustion engines. This was consistent with previous study that activated carbon led to reduce nitrogenated compound in diesel fuel [39], [40].

Meanwhile, high PAH would not be desirable as PAH was carcinogenic. Formation of PAH occurred from post-cracking reaction when reaction temperature and radiation time exceeds its limit.
Due to reaction limitation, when it exceeded radiation time of reaction, the reaction would proceed to produce PAH. It was supported by Lam et. al that at given degree of aromatization, condensation reaction of ring structure of aromatic compound occurred, which was eventually leading to formation of PAH [22]. In addition, heating mechanism promoted by microwave radiation energy resulted in uniform pyrolysis process, which would lead to formation of cyclo compound. Then, the dehydrogenation reaction increased in formation of aromatic compound in pyrolytic oil. Because of rapid reaction in microwave assisted pyrolysis added with reaction improvement by the presence of activated carbon and catalyst ZSM-5, aromatization of PAH has reduced and it formed more aromatic compound which was favourable.

On the other hand, monoaromatic and aliphatic compound were desirable. Existence of monoaromatic and aliphatic compound could enhance quality of pyrolytic oil as alternative fuel. In the formation of monoaromatic compound, role of catalyst in the microwave assisted pyrolysis process promoted decarboxylation reaction of oxygenated compound in non-phenolic fraction of pyrolytic oil, thus transformed oxygenated hydrocarbon compound in APS to aromatic compound [41]. In addition, dehydration, aromatization and alkylation reaction also occurred when Brønsted acid catalyst such as catalyst ZSM-5 was used [38]. Another study pertaining to microwave assisted pyrolysis was highlighted by Narula et. al, which stated that pyrolytic derived from dried paint sludge had no aromatic compound. Non-existence of aromatic compound in pyrolytic oil would affect fuel properties in terms of combustion energy by lowering down the calorific value [42]. Thus, catalytic microwave assisted pyrolysis would be very advantageous as it was time and energy-saving and produced high monoaromatic compound which contributed to high calorific value of pyrolytic oil.

Formation of aliphatic compound was found to be evident in pyrolytic oil of OFAT condition. Due to presence of catalyst ZSM-5 and activated carbon in microwave assisted pyrolysis of APS, alkylation, dehydration and decarboxylation reactions also occurred [38]. Alkylation, dehydration and decarboxylation reactions promoted increase of aliphatic compound in pyrolytic oil in the form of conjugated diene and intermediate denophile. It was also agreed by Bolotov et. al that presence of catalyst ZSM-5 in microwave assisted pyrolysis improved formation of aliphatic compound [43].

**Conclusion**

In a nutshell, presence of activated carbon and catalyst ZSM was capable to be used in order to produce pyrolytic oil. In this study, effect of activated carbon and catalyst ZSM can be seen by manipulating other factors such power level and radiation time as well. By using One-Factor-At-Time Analysis (OFAT), it was concluded that at 1000W power level, 30 minutes radiation time, 6% activated carbon loading and 1.5% catalyst loading, it was the best parameter to produce highest oil yield of 7.75% pyrolytic oil as compared to previous study, which was 0.875% pyrolytic oil produced with microwave-assisted pyrolysis of APS using catalyst only [17].

On the other hand, pyrolytic oil produced from OFAT via microwave assisted pyrolysis of automotive paint sludge (APS) with activated carbon and catalyst were also analyzed to determine the physical and chemical properties of the pyrolytic oil. By all, it was summarized that physical properties density, viscosity and calorific value were improved as compared to process without both activated carbon and catalyst.

As for chemical properties of the pyrolytic oil, it was evidenced that monoaromatic and aliphatic compound, which were desired in the pyrolytic oil, have increased compared to previous study. Meanwhile, undesired compound, which includes oxygenated, nitrogenated and polycyclic aromatic compounds were reduced and lead to enhance the quality of pyrolytic oil.

In conclusion, microwave assisted pyrolysis showed a huge potential in treating hazardous waste such as automotive paint sludge (APS) by using combination of activated carbon and catalyst ZSM-5.
As such by adding activated carbon and catalyst, process of microwave assisted pyrolysis was enhanced and improved quality of pyrolytic oil.

Acknowledgement

Authors would like to thank the financial support by Universiti Teknologi MARA (UiTM) through Geran Inisiatif Penyelidikan (GIP), Institute of Research Management and Innovation (IRMI), Faculty of Chemical Engineering and UiTM for research facilities.

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