

The Effect of HCl Concentration on The Activation of Bentonite as A Catalyst in The Pyrolysis Process of Polypropylene (PP) Plastic Waste at The Integrated Waste Management Facility of Asari Cilegon

Endang Suhendi^{1*}, Heri Heriyanto¹, Muhammad Khoirul Anam², Anida Aulia¹, Rizki Amalia Putri¹, Wardalia¹

¹Department of Chemical Engineering, Faculty of Engineering, Universitas Sultan Ageng Tirtayasa, Jl. Jenderal Sudirman KM 3, Kotabumi, Subdistrict of Purwakarta, City of Cilegon, Province of Banten, Indonesia

²Department of Government Relations and Circular Economy, PT Chandra Asri Petrochemical Tbk, Jl. Raya Anyer – Sirih KM 123, City of Cilegon, Province of Banten, Indonesia

* Corresponding author Email: endangs.untirta@gmail.com

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ABSTRACT

Pyrolysis is the process of heating a substance without the presence of oxygen, causing the decomposition of plastic materials (polymers). Pyrolysis is used as an alternative to reduce the amount of plastic which is a source of environmental problems. This research aimed to determine the effect of HCl concentration on bentonite catalyst activation on the liquid product yield of pyrolysis of polypropylene (PP) plastic waste and to determine the characteristics of the liquid pyrolysis products. This research was carried out in several stages including raw material preparation, catalyst activation, and pyrolysis process. In bentonite catalyst activation, the HCl concentration was varied to 0.25, 0.50, and 0.75 M. The results showed that the highest liquid product yield was obtained at a concentration of 0.50 M, namely 76% with a product density range of 0.7452-0.762 g/ml. The heating value of the liquid product at 0.50 M HCl activated bentonite was 6806.48 cal/g. Through GC-MS analysis, the liquid product contained 2,4-Dimethyl-1-heptene with an area of 8.21% and a retention time of 3.626. Based on XRD analysis, the bentonite contained minerals of montmorillonite, kaolinite, and quartz.

Keywords: Bentonite, GC-MS, Polypropylene, Pyrolysis, XRD

1. INTRODUCTION

Plastic plays an important role in everyday human life due to its efficiency in several sectors such as packaging, construction, automotive, electronics, agriculture, and others. So far, the use of plastic in modern life has become increasingly widespread and cannot be avoided, resulting in an increase in global plastic production every year from various industries and households so that it ends up as rubbish/waste. Waste is the remains of human activities or activities, both domestic and industrial.

Based on data from the Environment and Forestry Service of Banten Province, the Banten Province contributes waste of 7,190 tons/day or the equivalent of 2.62 million tons/year. Cilegon City contributes wastes of 83.16 thousand tons/year. Data on the origin of waste in the National Waste Management Information System (SIPSN) in 2022 shows that the waste comes from households 60%, traditional market waste 15%, trade center waste 7.1%, public facility waste 5.7%, waste area 4.1%, office waste 3.9%, and others. Plastic waste in Cilegon City was about 16% or 13.3 thousand tons/year.

A high accumulation of plastic waste can cause environmental problems because plastic waste is difficult for microorganisms to decompose (Savira and Okik, 2018). The methods for processing plastic waste to reduce environmental problems include recycling into new plastic materials, pyrolysis, and Refuse Derived Fuel (RDF). Pyrolysis is one of the methods that can be used to convert plastic waste into an alternative energy source (fuel).

One factor that can influence the pyrolysis process is the presence of catalysts. The pyrolysis process with the addition of catalysts can speed up the degradation reaction and shorten long hydrocarbon chains so that they can be easily condensed into pyrolysis oil. One natural catalyst that can be used is bentonite. The pyrolysis process with a catalyst has been carried out by several researchers. Dewangga et al. (2019) conducted research on polystyrene plastic waste with a variation of weight percentages of bentonite catalysts. The results of the research showed that the highest catalyst mass percentage produced the highest yield of liquid product. To improve the bentonite catalytic properties, an activation process is carried out using an HCl solution. In the pyrolysis process of plastic waste, the concentration of the HCl solution in bentonite activation can affect the pyrolysis products.

The novelty of this research is the pyrolysis process by modifying the bentonite catalyst with different acid solution concentrations. This research aimed to determine the effect of HCl concentration on bentonite catalyst activation on the liquid product yield of the pyrolysis of polypropylene (PP) plastic waste and to determine the characteristics of liquid pyrolysis products.

2. MATERIALS AND METHODS

2.1 Raw Material Preparation

The polypropylene plastic waste (clear packaging plastic) was obtained from the Waste Bank - Faculty of Engineering, University of Sultan Ageng Tirtayasa. The collected plastic was washed and dried in the sun, then cut into small pieces measuring ± 2 cm.

2.2 Bentonite Activation

The catalyst used was natural bentonite originating from Jasinga, West Java Province. The bentonite was crushed and screened using a 60-mesh sieve, then activated using an HCl solution with concentrations of 0.25, 0.50, and 0.75 M. The ratio of bentonite to HCl solution was 3:7. The activation process was carried out by dissolving 171.5 g of bentonite in 400 ml HCl which was heated using a hot plate at a temperature of 70°C accompanied by stirring with a magnetic stirrer for 3 hours. Next, the activated bentonite was cooled to room temperature, and then the HCl solution was separated from the bentonite. Furthermore, the bentonite was rinsed using distilled water until its pH was 7. The bentonite that had been neutralized was then placed in an oven at a temperature of 100-110°C for 3 hours.

2.3 Pyrolysis Process

The prepared raw materials as much as 250 grams were then put into the reactor. Then, a catalyst of 5% was added. The reactor was closed and heated to a temperature of 300°C for 3 hours.



Fig. 1. Schematic of the Pyrolysis Equipment

Where:

1. Termometer of condenser
2. Reaktor
3. Burning furnace
4. Condenser
5. Termometer of reaktor
6. Valve for liquid product

2.4 Sample Analysis

The Bentonite catalyst was analyzed through XRD testing using a Bruker D8 Advance Eco instrument and GC-MS testing using an Agilent type 5977 B instrument in the Integrated Laboratory of Sultan Ageng Tirtayasa University. The density of liquid products was analyzed

through the ASTM D1298 standards in the Laboratory of Energy Processing and Production, University of Sultan Ageng Tirtayasa. The heating value of the liquid product was analyzed through the ASTM E1269 standards in the Laboratory of Forensics, Polri-Bogor.

3. RESULTS AND DISCUSSION

3.1 Bentonite Characteristics

Bentonite was activated using an inorganic acid compound in the form of HCl. The activation process aims to exchange balancing cations in the bentonite structure (Ca^{2+} , Na^+ , K^+) with H^+ ions originating from acid and to release Al, Fe, and Mg ions and other impurities from the structure lattice so that physically, the bentonite becomes active after being acidified. During the bentonite activation process, Al, Fe, and Mg dissolved in solution, then acid was absorbed into the bentonite structure and resulted in a series of structures having a larger surface area than the bentonite before activation. In this activation, there was an exchange of cations from mineral salts (Ca^+ and Na^+) in the bentonite interlayer layer with H^+ ions from the acid, followed by the dissolution of Al^{3+} ions and other metal ions such as Fe^{3+} from the bentonite lattice layer. Because of the dissolution of Al^{3+} ions, bentonite became negatively charged so that its absorption capacity would increase (Hymore, 1996). Cation exchange from bentonite was influenced by HCl concentration (Geraldina, et al, 2016). Increasing the HCl concentration in the bentonite activation process can result in the release of minerals from the bentonite structure. As more minerals were released, the overall bentonite mass might decrease. This can happen because some of the bentonite mass was initially minerals that were released or dissolved in the HCl solution. The decrease in bentonite mass after activation can be seen in Table 1.

Table 1. Bentonite Mass Comparison

HCl Concentration	Mass before activation (g)	Mass after activation (g)
0.25 M	171.5	154.35
0.50 M	171.5	151.06
0.75 M	171.5	149.55

The structural characteristics and crystallinity of bentonite were tested using an XRD instrument to determine the success of bentonite catalyst formation and to determine the other materials that makeup bentonite. X-ray diffraction or electromagnetic X-rays is an analysis used to determine crystallite material, both its structure and the phase of the material, such as identifying the crystallite structure (qualitative value) and phase structure (quantitative value) in a material by utilizing X-ray electromagnetic wave radiation (Munasir, et al., 2012). The working principle of XRD analysis is to diffract light that passes through crystal gaps or lattices that have a wavelength equivalent to the distance between atoms, which is around 1 Angstrom.

When an X-ray beam interacts with a material, some of the beam will be absorbed, then transmitted, and some will be scattered or diffracted, this scattering will be detected by XRD. The samples analyzed using XRD were samples of bentonite without activation (BNT-NA) and bentonite activated with 0.5 M HCl solution (BNT-0.5M). The results obtained from the XRD test can be seen in Figure 2.

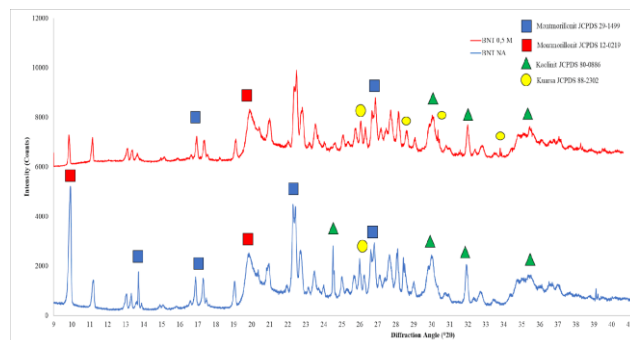


Fig. 2. Results of XRD Analysis of Bentonite Catalyst

Based on Figure 2, there are many peaks which are important parameters in explaining the diffraction pattern of the sample. XRD testing was carried out to determine the effect of the acid concentration used in the bentonite activation process on bentonite crystallinity. Figure 2 shows a change in the BNT-0.5M sample and the BNT-NA sample in the form of a decrease in the diffractogram intensity. The diffraction peaks at $2\theta = 9.87^\circ, 13.71^\circ, 22.30^\circ, 22.43^\circ, 22.72^\circ, 24.51^\circ, 25.99^\circ, 28.11^\circ, 28.42^\circ,$ and 31.94° . There is a decrease in intensity in the BNT-0.5M sample compared to the BNT-NA sample.

The use of HCl in the bentonite activation process caused a decrease in the diffractogram intensity of the BNT-0.5M sample. According to Siregar (2016), the decrease in diffractogram intensity and increase in peak width indicated that bentonite crystallinity was strongly influenced by acid activation. This shows that the process of forming an amorphous phase was caused by the decomposition of the montmorillonite structure. However, only part of the montmorillonite structure was decomposed because there were still montmorillonite peaks. Activation with HCl can break down mineral aggregates in bentonite and cause the formation of new compounds. BNT-0.5M experienced an increase in diffraction peaks at $2\theta = 26.03^\circ, 28.63^\circ, 30.34^\circ,$ and 33.81° . The use of an acid solution to activate the bentonite catalyst changed the Si/Al ratio which was a constituent of quartz compounds. The formation of quartz compounds due to an increase in acid concentration was proven in research conducted by Rahman et al (2022). The research showed that the XRD spectra of H_2SO_4 -activated bentonite experienced the formation of quartz compounds as evidenced by the addition of quartz compound peaks. The number of quartz compound peaks was twice as high in 3M H_2SO_4 activated bentonite compared to unactivated bentonite. Other research conducted by Vukovic et al (2006) showed that increasing the HCl concentration during

bentonite activation resulted in a narrowing of the quartz compound peak in the XRD spectra.

Based on the XRD spectra results of bentonite before the activation process, the peaks tend to be the same as bentonite after activation. This indicates that the bentonite sample has the same phase (Sibarani, et al. 2020). The XRD pattern (Figure 2) will be compared with JCPDS (Joint Committee on Powder Diffraction Standards) data to prove the similarity of the bentonite types.

Table 2. Values of 2 Theta (θ°) in Bentonite

Minerals	Sources	$2\theta^\circ$ (JCPDS)	$2\theta^\circ$ (BNT- NA)	$2\theta^\circ$ (BNT- 0.50M)
Montmorillonite	JCPDS 29-1499	17.8	17.26	17.31
		22.49	22.41	22.46
	JCPDS 12-0219	9.817	-	9.87
		19.87	19.82	19.85
Kaolinite	JCPDS 80-0886	24.51	24.49	24.51
		30.06	29.99	30.03
		31.98	31.94	31.93
		35.67	35.42	35.40
Kuarsa	JCPDS 88-2302	26.06	26.00	26.03
		28.85	28.41	28.63
		30.65	-	30.34
		33.04	-	33.81

Source: Data from JCPDS and Laboratory Analysis

Based on the data in Table 2, the diffraction patterns for BNT-NA and BNT-0.5M were in accordance with JCPDS data which showed that bentonite predominantly contained montmorillonite, kaolinite, and quartz minerals.

3.2 Effect of Heating Time on Temperature

The pyrolysis process is influenced by several factors such as heating time and temperature. The results of the comparison of time and temperature can be seen in Figure 3.

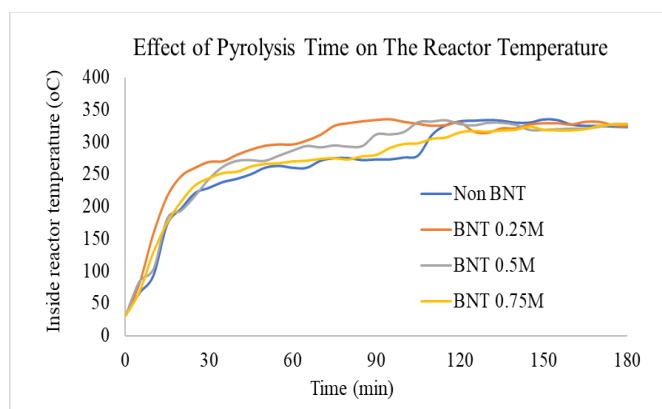


Fig. 3. Profile of the Relationship between Heating Time and Temperature

Based on the results obtained in Figure 3, it can be seen that the relationship between pyrolysis time and temperature was directly proportional. Increasing the time of the pyrolysis process was accompanied by an increase in the temperature of the pyrolysis process.

The increase in temperature was caused by the absorption of heat by the plastic raw material in the pyrolysis process. Plastic as a raw material will absorb heat and spread throughout the plastic surface in the reactor. Heat absorption over a longer time increased the temperature of the pyrolysis process. This caused the plastic to experience degradation of the constituent hydrocarbon chains into shorter hydrocarbon chains.

Plastic has thermal properties in the form of a melting point. The melting point is the point at which plastic begins to soften and change state to liquid. The melting point of polypropylene (PP) plastic is 160°C. Increasing the temperature in samples without a catalyst (Non-BNT) required a longer time. The addition of bentonite as a catalyst in the pyrolysis process can increase the heating rate. The presence of a catalyst in the pyrolysis process can help reduce the activation energy needed to start the pyrolysis reaction so that the pyrolysis process can take place more quickly. A concentration that is too low will cause incomplete formation of the active site, whereas a concentration that is too large will cause damage to the catalyst structure (Geraldina, et al, 2016).

3.3 Effect of Pyrolysis Time on Liquid Product Formation

Based on the results in Figure 4, the pyrolysis liquid product increased when the pyrolysis time increased. This was because the longer the pyrolysis time, the more evenly the contact between the heat and the raw material would increase, thereby increasing the degradation of compounds. The pyrolysis process involves cracking long-chain hydrocarbons into smaller molecules. The plastic would melt and change phase to liquid which was then heated to form a gas, then cooled and condensed to form a pyrolysis liquid with the highest yield of 76% with a pyrolysis time of 3 hours. This research was in accordance with research conducted by Arini (2022) reporting that the pyrolysis of polypropylene (PP) waste resulted in a yield of 76% at a temperature of 500°C.

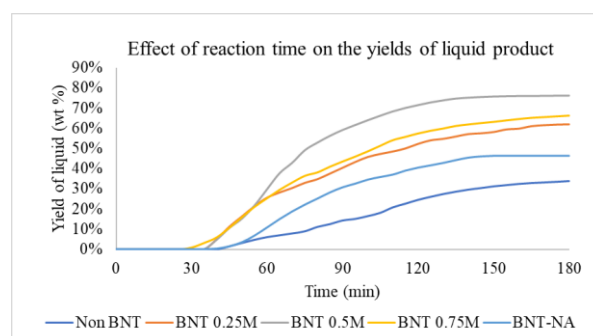


Fig. 4. Effect of Time on Liquid Product Formation

Based on Figure 4, the addition of a catalyst affected the time and formation of liquid products. The addition of an activated bentonite catalyst can accelerate the initial formation of pyrolysis liquid products and increase the yield of pyrolysis liquid products. In the pyrolysis using a BNT-0.75M catalyst,

the liquid product was first formed at minute 30. Meanwhile, in the pyrolysis without a catalyst (Non-BNT), the liquid product was first formed at minute 45. This shows that the bentonite catalyst influenced the speed at which the liquid product was first formed. Apart from that, the addition of a bentonite catalyst can accelerate the increase in yield of pyrolysis liquid products. The presence of a catalyst in the pyrolysis process functioned to accelerate the degradation reaction and shorten the hydrocarbon chain. This was because catalysts helped in the formation of free radicals which were formed due to the breaking of carbon chain bonds, thereby breaking long hydrocarbon chains into shorter hydrocarbon chains (Pratiwi & Wiwiek, 2019). The presence of a catalyst increased the temperature and reduced the reaction time. Apart from that, catalysts improved the quality and quantity of pyrolysis results (Budsareechai, S. et al, 2015). The active site provided by the catalyst can generate heat (Effendi, et al, 2021) so that the reactant molecules, in this case, the polypropylene branch chains, will easily be broken down into small parts which will then evaporate and be condensed into a liquid product. Research on the effect of catalysts on the time and yield of liquid pyrolysis products of polystyrene (PS) plastic waste conducted by Trisnaliani et al (2021) showed that adding 1 hour of catalyst time produced a yield percentage of 85.92%.

3.4 Effect of HCl Concentration in Bentonite Activation Process on Pyrolysis Products

The bentonite was activated using HCl with concentrations of 0.25, 0.50, and 0.75 M. With the same catalyst mass, the pyrolysis products were obtained as shown in Figure 5.

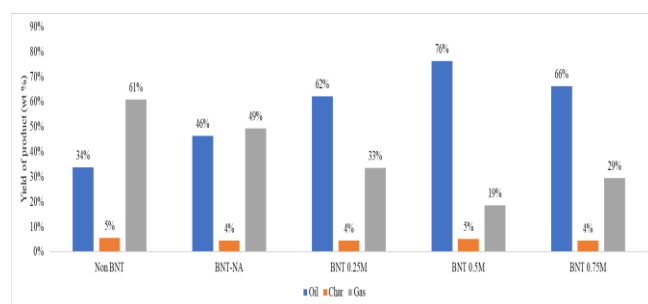


Fig. 5. Effect of HCl concentration on yield

Pyrolysis with activated bentonite provided higher liquid product yields compared to pyrolysis without a catalyst. Based on Figure 5, the liquid product yield reached 76% at BNT-0.5M. The increase in liquid product yield was almost 100% compared to the pyrolysis process without a catalyst. This was because the speed of product formation by BNT-0.5M was faster than that by the catalyst activated at concentrations of 0.25 M and 0.75 M. By activating using acid, bentonite contributed to an increase in liquid products while decreasing gas and solid products because the activation process opened the pore structure of the bentonite catalyst. By opening the pore structure of the

catalyst, the surface area increased. The highest liquid product yield was found in the BNT-0.5 M sample because an acid concentration of 0.5 M resulted in a catalyst with more surface area than the other concentrations. The decrease in liquid product yield occurred at BNT-0.75M. Increasing high concentrations caused catalyst deactivation. High HCl concentrations caused overactivation or deactivation of the bentonite catalyst in the pyrolysis process.

The liquid product resulting from pyrolysis in this study was compared to that in previous research conducting pyrolysis of PP (100%) without a catalyst, as shown in Table 3.

Table 3. Comparison of Yield of Liquid Products

Samples	Yield (%)	Jahiding et al. (2020)	Suhendi et al. (2023)
Non-BNT	34		
BNT-NA	46		
BNT-0.25M	62	16% (450°C) 64% (500°C) 70% (550°C)	63.51% (350°C)
BNT-0.50M	76		
BNT-0.75M	66		

Jahiding et al (2020) conducted research on the pyrolysis of 1 kg of PP plastic waste using temperature variations of 450, 500, and 550°C with a heating rate of 15°C/minute. The results of the previous research (Jahiding et al., 2020) showed that the yield at the lowest temperature was less than half the yield in this study. Apart from that, the yield at BNT-0.5M was still superior compared to the yield at a temperature of 550°C. This means that research using bentonite as a catalyst can reduce the energy required during the pyrolysis process. This was because the higher the pyrolysis temperature, the more energy was required (Suhendi, et al, 2023). Research conducted by Suhendi et al. (2023) with a feed amount of 100 kg and a pyrolysis time of 9 hours resulted in a yield of 63.51%. The research by Suhendi et al. (2023) resulted in a higher percentage compared to this research without a bentonite catalyst. Wicaksono and Arijanto (2017) explained that increasing the pyrolysis temperature can make it easier to break down the chains that make up the polymer in plastic so that the resulting yield is greater.

3.5 Characteristics of Pyrolysis Liquid Products

The pyrolysis products obtained were subjected to GC-MS, density, and heating value tests. Identification using GC-MS is used to determine the components or compounds contained in liquid products. A chromatogram of a propylene plastic pyrolysis sample using 0.50 M HCl-activated bentonite (BNT-0.50M) is shown in Figure 6.

4. CONCLUSION

Based on research that has been carried out, the highest liquid product yield was obtained at a concentration of 0.50 M HCl, namely 76% with a product density ranging from 0.7452-0.762 g/ml. The heating value of the liquid product at 0.50 M HCl activated bentonite was 6806.48 cal/g. Based on the GC-MS test, the liquid product contained the compound 2,4-Dimethyl-1-heptene with an area of 4.89% and a retention time of 3.626. Through XRD analysis, the bentonite contained minerals of montmorillonite, kaolinite, and quartz. This research can be continued by varying the pyrolysis temperature, activation time, type of activation solution, and catalyst particle size.

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