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Effects of cobalt oxide catalyst on pyrolysis of polyester fiber

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Abstract−Polyester fiber is a high-molecular-weight compound made from fossil fuels and is used in various synthetic fiber manufacturing processes. In this study, we performed non-catalytic and catalytic pyrolysis experiments using cobalt oxide as a catalyst to recover energy from polyester fiber. The experiment was carried out between 500-900 °C in the presence of N_2. Amount of oil formation was the highest at 600 °C in non-catalytic pyrolysis and oil formation of catalytic pyrolysis was the highest at 500 °C. In both non-catalytic pyrolysis and catalytic pyrolysis, gas content was increased and char was decreased with increasing temperature. A marked difference was observed when the catalyst was used; the formation of char was suppressed and oil and gas yields increased. In the catalytic pyrolysis oil, benzoic acid compounds accounted for the largest proportion (16.15 wt%) at 900 °C, but polycyclic aromatic hydrocarbons and phenols were not observed. Benzoic acid is an important precursor material used to synthesize other organic substances, such as phenol and caprolactam. The non-condensable gas content increased from 11.55 wt% to 22.39 wt%, with increasing temperature. In particular, H_2 gas yield was 4.44 wt% at 900 °C. Therefore, by using catalytic pyrolysis, high value-added chemicals such as benzoic acid compounds and H_2 gas can be recovered at high yield at 900 °C from the polyester fiber. Consequently, unlike the existing treatment methods, the environmental impact of plastics can be reduced by catalytic pyrolysis.

Keywords: Waste Treatment, Pyrolysis, Plastic Waste, Thermochemical Process, Reusable Energy, Catalytic Process

INTRODUCTION

Plastics have become essential materials in daily life because they are light, inexpensive, and convenient [1,2]. The production of plastics has grown rapidly in recent decades owing to their wide application in several sectors, such as packaging, building, automotive, electric and electronics, and agriculture [3,4]. Polyesters, such as polyethylene terephthalate (PET) with semicrystalline structure, are the main synthetic polymers for fiber production [5]. Polyester and other synthetic fibers have major advantages of high modulus and strength, stiffness, stretch, wrinkle and abrasion resistance, convenient processability [6]. Owing to these properties and low production costs, polyester fiber is one of the most widely used synthetic plastic fibers in apparel industries and contributes to more than 75% of global synthetic fiber production [7], including clothing fibers, furniture covers, blankets, waterproof sheets, industrial ropes, belts, tent fabrics, films, and filters [8,9].

Polyester fibers harm the environment during and after use. Microplastics are released from clothes made of polyester during washing. Microplastics enter sewage treatment plants but cannot be treated and eventually reach the sea. Microplastics are non-biodegradable and break down into fine particles that float [10,11]. These microplastics adversely affect marine ecosystems, become accumulated in the bodies of marine organisms, and finally bioaccumulate in the human body as they move up the food chain [12]. The total production of different man-made fibers was 71.6 million tons last year, with polyester accounting for three-quarters of the total [7]. Because of its wide use in the textile industry, polyester fiber is one of the most commonly found microplastics in the seas and oceans.

Plastics are typically treated by incineration, recycling, and landfilling [13]. However, because polyester is used in combination with other fibers or materials [14,15], it is difficult to separate polyester fibers from those materials, making it hard to recycle them [16]. The recycling efficiency of polyester fibers is less than 1% on average [17]. In addition, treating plastics, such as polyesters, through landfilling can pollute the environment. Coastal landfills are a source of plastic pollution that affects terrestrial and marine ecosystems. Eroding landfills potentially pollute seas and contribute to climate change [12]. In addition to the potential environmental damage caused by plastic waste in the landfills, the decreasing space for landfills requires the implementation of alternative options for the disposal of plastics [18]. Other methods such as incineration generate air pollutants, including hydrogen chloride, dioxin, cadmium, and fine particulate matter [19]. Polyester fiber is made of petroleum derivatives and contains abundant carbon, so it is desirable to reuse it for waste management and carbon reuse.

Pyrolysis is a feasible and eco-friendly method for plastic waste disposal [20]. It helps reduce waste volume. Pyrolysis is a one of the waste treatment methods that is applicable to various wastes and

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mixed wastes [21,22]. Plastic waste, including polyester fiber waste, can be valorized into gas, oil, and char via pyrolysis; they can be used as fuel or raw material for plastic production, according to their characteristics [23].

Catalytic pyrolysis can be selected to increase or decrease the number of pyrolysis products or to improve their quality [24-26]. In this study, we sought to reduce volatile organic compounds (VOCs) in pyrolytic oil using a cobalt oxide catalyst [27]. In addition, we aimed to reduce the environmental impact of plastics and produce high value-added chemicals through polyester pyrolysis, unlike the existing treatment methods. Various studies have been conducted on catalytic pyrolysis of plastics, such as improving oil yield through catalytic pyrolysis of plastic waste [28-30] or reducing pollutants in pyrolytic products using catalysts [25,31,32]. However, there no research has been conducted for qualitative/quantitative analysis of three-phase products produced by pyrolysis of polyester fibers to present the yield of useful substances. This study will contribute to the approach of converting polyester fibers into useful substances for sustainable resource circulation. We conducted the study using commercially available polyester product, wherein pyrolysis was carried out under various conditions, and the differences in each condition were analyzed.

MATERIALS AND METHODS

1. Feedstock and Catalyst Characterization

A polyester fiber was provided by a printing house located in Suwon, Gyeonggi Province, Republic of Korea. The polyester fiber was cut into pieces (1×45 cm) and sample was dried at 60 °C overnight to remove moisture before use as feedstock. The characteristics of the feedstock were analyzed by proximate analysis, ultimate analysis, and thermogravimetric analysis (TGA). The Co_3O_4 catalyst (CAS No. 1308-06-1), with a particle size <50 nm and (Brunauer, Emmett, and Teller) BET surface area of 40-70 m^2/g, was purchased from Sigma-Aldrich.

Moisture, volatility, ash, and fixed carbon content were measured using proximate analysis. Moisture was measured by heating the polyester fiber at 105 °C for 24 h, and the content of volatile substances was measured by heating at 405 °C for 1 h under closed conditions. The ash content was measured by heating at 750 °C for 1 h. The fixed carbon content was determined by subtracting the values of moisture, volatilization, and ash contents from the weight of the raw material.

The C, H, N, and S contents were measured by ultimate analysis, using a FlashSmart 2000 elemental analyzer manufactured by Thermo Scientific (Waltham, MA, USA).

TGA was performed using a STA449 F3 thermal analyzer (NETZSCH, Selb, Germany) to raise the polyester fiber at 10 °C/min from 30 °C to 1,000 °C in the presence of N_2 (flow rate: 60 mL/min).

2. Pyrolysis Reactor Setup and Procedure

We performed both non-catalytic and catalytic pyrolysis in the presence of N_2. The feed was loaded into a quartz tube (length: 0.6 m; outside diameter: 25 mm; inside diameter: 21 mm) using quartz wool and placed in the center of the pyrolysis reactor. The quartz tube was purged with N_2 gas, and the gas was flowed at 50 mL/min using a mass flow controller during the reaction. The gas was maintained at approximately 80 °C, using heating tape, before it entered the quartz tube. The experimental temperature was set from 500 °C to 900 °C based on the TGA results of the polyester fiber, and the heating rate was 10 °C/min. Ice bath (−1 °C) and dry ice bath (−60 °C) were used to collect the pyrolytic oil. Then, the pyrolytic oil was dried in a drying oven at 40 °C. The outlet of the reactor was connected to the micro-GC.

Catalytic pyrolysis was performed by loading 0.05 g (5 wt%) of the catalyst along with feed in the quartz tube (i.e., in-situ configuration). Other conditions were the same as those used for the non-catalytic experiment.

3. Product Analysis

A gas chromatograph/mass spectrometer (GC/MS; GC model: 8890; MS model: 5977 B; Agilent Technologies, Santa Clara, CA, USA) equipped with an HP-5MS column (Agilent Technologies; diameter: 0.25 mm; length: 30 m; film thickness: 0.25 μm) was used to analyze the composition of the pyrolytic oil and the concentration of each constituent; helium was used as the carrier gas. 1 μL of pyrolytic oil was injected into the GC/MS and heated to 280 °C. The initial temperature of the oven was set at 50 °C for 5 min and then increased to 280 °C at a rate of 10 °C/min. The final temperature was maintained for 5 min. The AUX temperature was 300 °C, and the m/z range was set to 50-500 amu. The composition of the oil was analyzed using the NIST program. For an internal standard, 5 ng/mL of 5-methylfurfural was used to calculate concentrations of the identified species.

Pyrolytic gases were measured using a Fusion Gas Analyzer micro-GC manufactured by INFICON (Bad Ragaz, Switzerland). The micro-GC was composed of module A with a silica PLOT column (Rt-Msieve 5A, Restek (Bellefonte, PA, USA)) and module B with a non-polar PLOT column (Rt-Q-BOND, Restek). UHP argon and UHP helium were the carrier gases for each column. The pyrolytic gas entered the column through an injector (injector temperature of 90 °C). In module A, the temperature was maintained at 50 °C for 40 s, increased to 100 °C for 50 s, and then maintained for 40 s. The temperature in module B was maintained at 50 °C for 30 s, increased to 110 °C for 60 s, and maintained at 110 °C for 40 s. The temperature of the thermal conductivity detector was set at 70 °C.

RESULTS AND DISCUSSION

1. Feedstock Characterization

Fig. 1 shows the thermal degradation patterns of the polyester fiber obtained by TGA, which was performed in the presence of N_2. Between 500 °C and 900 °C, approximately 80 wt% of the polyester fiber sample was decomposed; however, approximately 19.45 wt% of the polyester fiber sample was not decomposed until 900 °C, which indicates that 19.45 wt% of the polyester fiber was made up of fixed carbon. This result is consistent with the proximate analysis (Table 1) of the polyester fiber.

Table 1 shows the proximate and ultimate analyses of the polyester fiber. It consisted of volatile matter (74.40 wt%), fixed carbon (19.11 wt%), moisture (1.39 wt%), and ash (5.11 wt%). The results of the TGA and proximate analyses were congruent. The polyester fiber

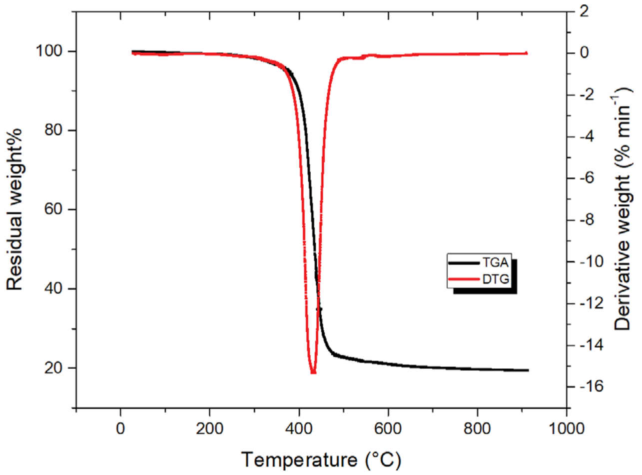

Fig. 1. Residual mass% and derivative weight profiles of the polyester fiber.

Table 1. Results of ultimate and proximate analysis of the polyester fiber

		Content
Ultimate analysis (wt%)	C	59.09
	H	4.52
	O^a	36.13
	N	0.26
	S	-
Proximate analysis (wt%)	Moisture	1.39
	Volatile	74.40
	Fixed carbon	19.11
	Ash	5.11

^{a}Different by ultimate analysis result

had approximately 59.09 wt% carbon content and 36.13 wt% oxygen content. Thus, the polyester fiber had high carbon and oxygen content (Table 1). The structural formula of polyester is $(C_{10}H_8O_4)_n$, and it has approximately 62.5 wt% carbon content and 33.3 wt% oxygen content. This is similar to the results obtained from the ultimate analysis. It has been estimated that 0.26 wt% of nitrogen is derived from a small amount of ink or coating agent contained in the polyester fiber.

2. Effects of Cobalt Oxide Catalyst on Pyrolytic Products of Polyester Fiber

Fig. 2 shows the mass balance of pyrolysis of the polyester fiber with and without the cobalt oxide catalyst. At all temperatures, regardless of the catalyst, the total non-condensable gas yield increased as the temperature increased. This is because high temperatures promote thermal cracking, which shifts the carbon distribution from wax and char to pyrolytic gas. This tendency was more pronounced in catalytic pyrolysis. The yield of volatile was calculated by subtracting the weight of other pyrolytic products (gas, oil, wax, char) from the weight of raw feed. More volatile compounds and non-condensable gases were observed in catalytic pyrolysis than in non-catalytic pyrolysis, and the proportions of char and wax were reduced. Char is a solid product of pyrolysis, and wax is a heavy oil composed of paraffins, olefins, aromatics, and heavy compounds (length of carbon chain: above C_{20}) [33]. In addition, in the non-catalyst pyrolysis, the highest yield of oil was obtained at 600 °C, but in the catalytic pyrolysis, the highest yield was obtained at 500 °C. This indicates that the cobalt oxide catalyst assisted in thermal cracking.

3. Analysis of the Pyrolytic Solid

In both catalytic and non-catalytic pyrolysis, the rate of char production decreased with increasing temperature. The char content produced from non-catalytic pyrolysis decreased from 21.57 wt% at 500 °C to 19.21 wt% at 900 °C, and the char content produced from catalytic pyrolysis decreased from 20.10 wt% at 500 °C to 17.82 wt% at 900 °C (Fig. 2). It was confirmed that the use of the catalyst suppressed the formation of char. In addition, the char yield was affected by temperature. When the temperature was increased above 400 °C, C-H and -OH bonds were cleaved. Therefore, more volatile compounds were produced in the feed [34]. As a result, the char content decreased as the temperature increased.

The ultimate analysis of the char generated at 500 °C by catalytic

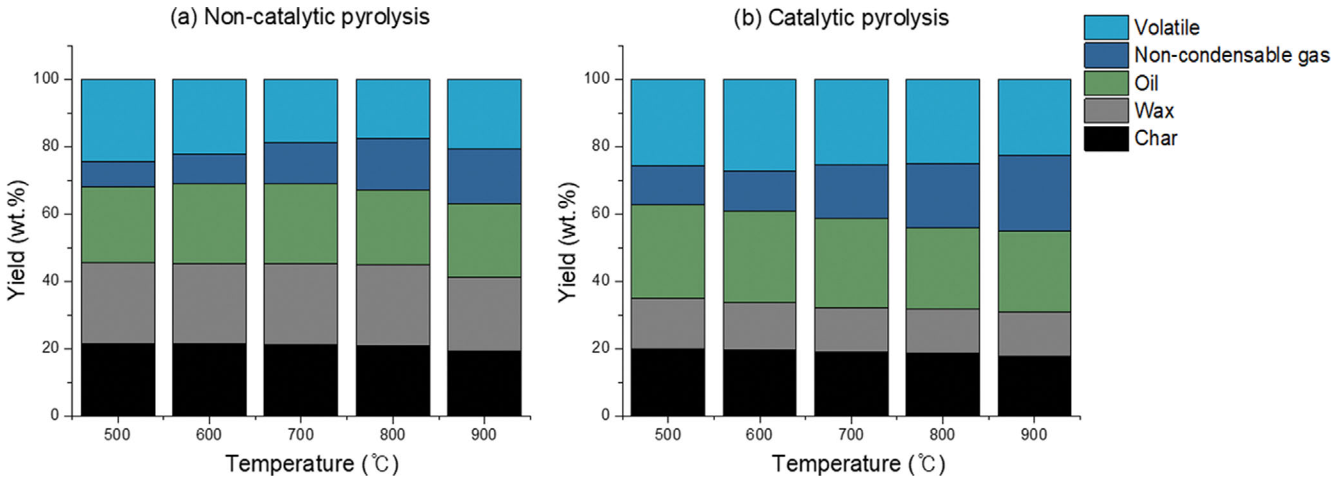

Fig. 2. Mass balance of the polyester fiber after pyrolysis (a) without catalyst and (b) with a cobalt oxide catalyst.

Table 2. Results of the ultimate analysis and heating values of char produced through non-catalytic pyrolysis and catalytic pyrolysis at 500 °C

		Non-catalytic pyrolysis	Catalytic pyrolysis
Ultimate analysis (wt%)	C	58.94	53.41
	H	2.8	2.47
	O	37.86	43.69
	N	0.4	0.43
	S	-	-
Heating value (MJ/kg)		19.72	16.76

and non-catalytic pyrolysis was also performed. In the ultimate analysis, the carbon ratio of the char produced by non-catalytic pyrolysis was smaller than that of the catalytic pyrolysis. Based on the results of the analysis, the heating value was calculated using the following equation [3]:

$$Q=151.2\ (C)+499.77\ (H)+45.0\ (S)-47.7\ (O)+27.0\ (N)$$

The heating values of char of non-catalytic and catalytic pyrolysis were 19.72 MJ/kg and 16.76 MJ/kg, respectively (Table 2). As mentioned, the catalyst decreased the generation of char. Therefore, the heating value of char of non-catalytic pyrolysis was more than that of char of catalytic pyrolysis. As the heating value of lignite is 18.29 MJ/kg, it was confirmed that char from non-catalytic pyrolysis can be used as fuel instead of lignite [34].

4. Analysis of the Pyrolytic Oil

The composition of pyrolytic oil was identified using GC/MS, and its constituents were classified into polycyclic aromatic hydrocarbons (PAHs), benzoic acid compounds, benzoate, phenol, polyphenyl compounds and others (Fig. 3). The pyrolytic oil exhibited various distributions for each temperature range. In non-catalytic pyrolysis, all six classifications were identified in the oil. Yield of the oil was highest at 600 °C. The yield of benzoic acid compounds, benzoate and phenol which are components of the pyrolytic oil, was also the highest at 600 °C. Notably, PAHs and polyphenyl compounds were not. The production of PAHs increased with increasing temperature (6.19×10^{-4} g to 8.46×10^{-4} g). Previous studies have also shown that the yield of PAHs increases as temperature increases [8,16].

In catalytic pyrolysis, PAHs and phenol were not produced, but only benzoic acid compounds, benzoate, and polyphenyl compounds were produced. The oil yield was similar across all temperature ranges. Benzoic acid compounds accounted for the largest proportion of the oil, and its yield was the highest at 900 °C (16.15 wt%).

A comparison of the catalytic and non-catalytic pyrolysis oils revealed that phenol and PAHs were not produced during catalytic pyrolysis. PAHs are formed from benzene, acetylene, and phenol. Benzene and acetylene, which form PAHs, phenol, are VOCs [35]. When benzene and acetylene are pyrolyzed together, high-molecular-weight PAHs are formed [36], and benzene affects the growth of PAHs as first aromatic ring [37,38]. Benzene reacting with OH produces phenol [39]. Radical reactions of phenol can produce PAHs [40]. Cobalt oxide is a catalyst that decomposes VOCs into non-condensable gases [27]. Therefore, PAHs and phenol are not formed in the catalytic pyrolysis because VOCs are consumed via cobalt oxide-catalyzed reaction. In addition, the yields of benzoic acid compounds, benzoate, and polyphenyl compounds showed the greatest differences in oil products. Benzoic acid, which accounts for the largest proportion, is an important precursor material used to synthesize other organic substances, such as phenol and caprolactam [41,42]. The synthesis of benzoic acid may cause undesirable side reactions and harmful environmental effects [42]. Therefore, if the catalytic pyrolysis method is used, the yield of benzoic acid compounds can be increased.

5. Analysis of the Pyrolytic Non-condensable Gases

Fig. 4 shows the yields of non-condensable gases obtained by the thermochemical conversion of the polyester fiber in the presence of N_2, at different temperatures (500-900 °C). The total non-condensable gas yield increased with increasing temperature in both non-catalytic pyrolysis and in situ catalytic pyrolysis. When the pyrolysis temperature was increased from 500 °C to 900 °C, the gas yield increased from 7.40 to 16.24 wt% in non-catalytic pyrolysis and from 11.55 to 22.39 wt% in in-situ catalytic pyrolysis. This could be due to the enhanced thermal cracking of condensable gas via

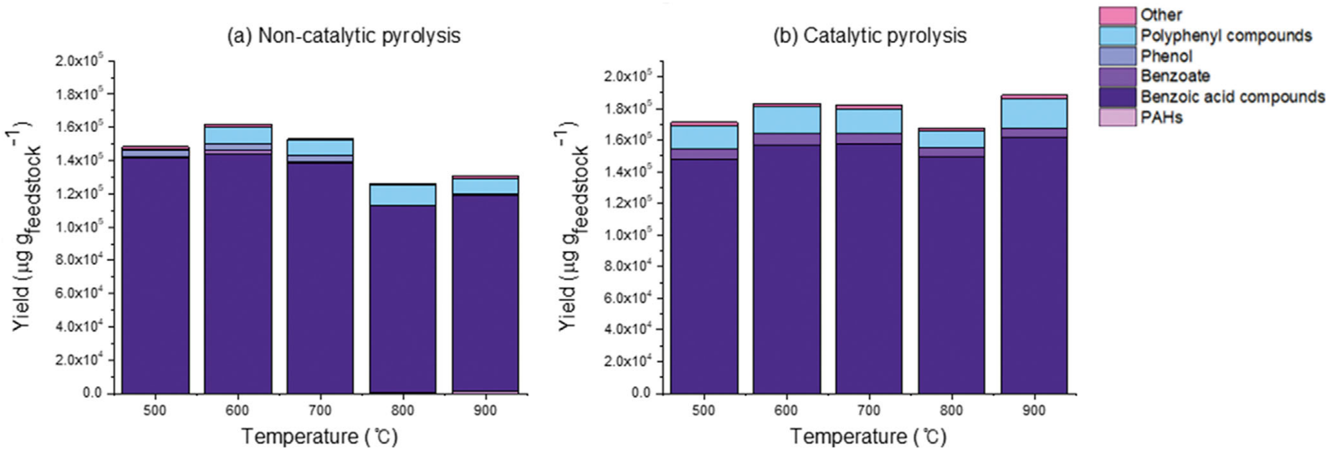

Fig. 3. Yields of pyrolytic oil compounds produced by (a) non-catalytic pyrolysis and (b) catalytic pyrolysis of the polyester fiber.

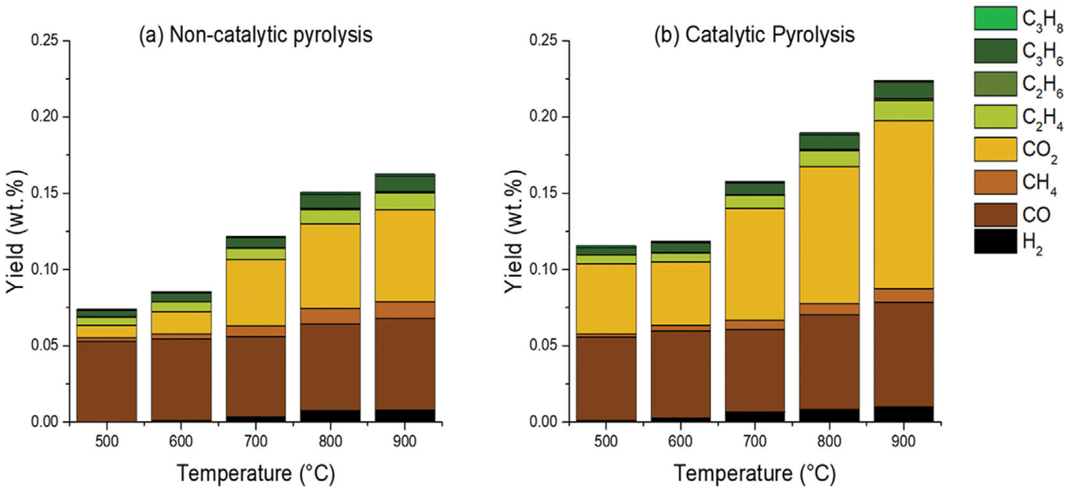

Fig. 4. Yields of pyrolytic gas compounds produced from polyester fiber by (a) non-catalytic pyrolysis and (b) catalytic pyrolysis.

heterogeneous reactions occurring in the gas-solid phase and homogeneous reactions occurring in the gas phase as the temperature increases [43]. At all temperatures, the gas yield was higher during the in situ catalytic pyrolysis than during the non-catalytic pyrolysis. In addition, the increase in the total non-condensable gas yield obtained through the thermochemical conversion of the polyester fiber in catalytic pyrolysis was greater than that in non-catalytic pyrolysis. In non-catalyst pyrolysis, as the temperature increased, the total non-condensable gas yield increased by only 8.84 wt%, but in the catalytic pyrolysis, it increased by 11.84 wt%. This is because the cobalt oxide catalyst promoted the thermal cracking of the feedstock. Moreover, cobalt may provide Lewis acid functionality [44]. This further enhances the cracking capacity, leading to the formation and release of more volatile compounds at lower temperatures [44].

Hydrogen gas, a gas species produced through pyrolysis, is one of the most promising energy carriers and is considered to be the cleanest fuel. This is because only water is produced during its combustion [45]. When burning fossil fuels, various pollutants, such as CO_2, particulate matter (PM), and ozone are generated [46]. Burning H_2 as a fuel generates fewer pollutants than burning fossil fuel. In addition, it generates more energy on a mass basis than fossil fuels, such as coal, gasoline, and methane [34]. Pyrolysis is advantageous over other processes for the production of gaseous H_2 because of its simplicity and fuel adaptability [47]. Fig. 5 shows the yield of H_2 gas according to pyrolysis temperature during catalytic and non-catalytic pyrolysis. The H_2 gas yield increased with increasing pyrolysis temperature in both non-catalytic and catalytic pyrolysis. This is because higher pyrolysis temperatures promote the decomposition of vaporized species released from the feedstock during the pyrolysis process. The H_2 gas yield was higher in the catalytic pyrolysis than in the non-catalytic pyrolysis at all temperatures. This is because the cobalt oxide catalyst accelerates the dehydrogenation reaction. Cobalt oxide exhibits good performance with high selectivity for H_2 [48]. However, as the reaction proceeded, the cobalt oxide catalyst reacted with H_2 and was reduced to Co [49]. Nevertheless, the reason why H_2 production of catalytic pyrolysis was higher than non-catalytic pyrolysis was that Co affects H_2 production as catalyst. According to research results, adding a catalytic active element such as cobalt to biomass-based char has a huge influence on H_2 production. It was also found to have an effect on heavy compound cracking and reforming [50]. Therefore, even if the catalyst is reduced, H_2 production continues to increase.

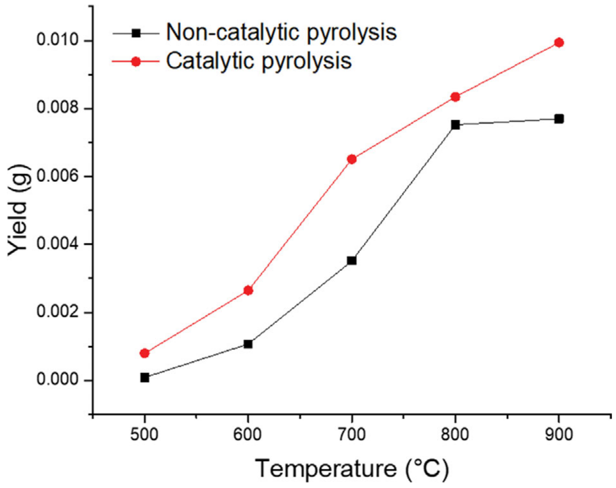

Fig. 5. Comparison of H_2 gas yield in catalytic pyrolysis and non-catalytic pyrolysis performed at different temperatures.

Ethylene and propylene yields increased during catalytic pyrolysis. They are widely used in the production of various chemical intermediates and polymers; worldwide, ethylene consumption exceeded 150 million tons per year in 2017 [51] and propylene production exceeded 120 million tons per year in 2017 [52]. Currently, ethylene is primarily produced through steam cracking of ethane and naphtha [53]. Propylene is conventionally produced as a byproduct of ethylene from steam cracking or as a byproduct of gasoline in fluid catalytic cracking in refineries [54]. In this study, the ethylene yield was 5.94 wt% and propylene yield was 4.99 wt% of the total non-condensable gas mass (in-situ catalytic pyrolysis, 900 °C). Thus, pyrolysis can be a useful means of treating waste and producing high-value-added chemicals, such as ethylene, propylene, and H_2 gas.

CONCLUSIONS

Polyester fiber is a synthetic fiber that is used in widely in industry and has gained the attention of the researchers because of its environmental impact. Pyrolysis is the preferred method for treating polyester fiber because waste can be reduced and converted into high-value products, such as benzoic acids compounds and H_2 gas. In this study, catalytic pyrolysis using a cobalt oxide catalyst is proposed as a method for increasing the production of such high-value-added materials. The results indicate improved pyrolytic oil and non-condensable gas yields. PAHs and phenol content in pyrolytic oil was reduced. In addition, the yields of H_2, ethylene, and propylene, which are non-condensable gases, also improved. During the catalytic pyrolysis, 16.15 wt% of benzoic compounds were recovered at 900 °C. The gas content increased from 11.55 wt% (500 °C) to 22.39 wt% (900 °C). H_2 gas yield was 4.44 wt% at 900 °C, and it was used to make combustible gas. Also, ethylene yield was 5.94 wt% and propylene yield were 4.99 wt% of the total non-condensable gas mass at 900 °C. H_2 gas is a next-generation clean energy fuel, and ethylene and propylene are used in the production of various chemical intermediates and polymers.

This study demonstrates that the use of transition metal oxide catalysts, such as cobalt oxide supported for the pyrolysis of polyester fibers (and potentially other soluble fibers and plastic materials), could contribute to renewable and eco-friendly waste treatment. Based on this study, it will be possible to improve the yield of H_2 and useful substance yield through co-pyrolysis with biomass or other plastic waste. Furthermore, this study should contribute to innovative approaches to providing promising solutions to H_2 production and waste treatment. The stability of cobalt oxide catalyst during pyrolysis would be a future work that can help further enhance the catalytic pyrolysis process efficiency [55,56].

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