



Synthesis of biofuel via catalytic fast pyrolysis of wood-plastic composite over low-cost catalysts

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ABSTRACT

Thermogravimetric (TG) and pyrolyzer-gas chromatography/mass spectrometry (Py-GC/MS) analyses were performed to know the feasibility of low cost catalysts, γ -Al₂O₃, spent fluid catalytic cracking (FCC) catalyst, natural zeolite, to the pyrolysis of wood-plastic composite (WPC). Notably, the respective decomposition temperature of polyethylene and polypropylene in WPC was lowered with the use of low-cost catalysts during the catalytic fast pyrolysis (CFP). Furthermore, the lowest activation energy (E_a) of the CFP, obtained by the Ozawa method, was with the use of natural zeolite (127.5 kJ/mol), followed by the ones with the use of spent FCC catalyst (133.3 kJ/mol) and γ -Al₂O₃ (145.0 kJ/mol), respectively. In addition, the CFP over natural zeolite was less effective in terms of decreasing the production of low-quality hydrocarbons, paraffins and olefins having a wide carbon number distribution, and oxygenates compared to that over spent FCC catalyst and γ -Al₂O₃, respectively, due to the small pore size of natural zeolite. However, the largest amount of value-added aromatic hydrocarbons was produced in the CFP over natural zeolite among all the catalysts due to the high and strong acidity of natural zeolite.

Introduction

The development of science and technology has enriched human life, and plastic has brought many changes in our daily life. However, a large amount of plastic waste enters the environment, and this waste is difficult to decompose naturally and causes problems such as microplastic accumulation and fossil fuel depletion [1]. In addition, the shortage of fossil fuels is accelerating due to the increased use of petroleum for plastic synthesis, and climate change is also accelerating due to the increased concentration of carbon dioxide in the atmosphere owing to the excessive use of fossil fuels driven by this plastic synthesis [2,3].

To cope with the problems related to plastic usage and waste, global organizations, such as the international panel on climate change (IPCC), have been emphasizing practical measures to slow down global warming. In particular, these organizations implement measures to reduce the usage of petrochemicals and find new renewable sources of energy, and

frame energy usage reduction policies. One of the technologies that aligns with these measures is the production of fuel and chemical raw materials from waste plastics and waste biomass via thermal conversion technologies, such as torrefaction, pyrolysis, gasification, and so on [4–8].

Wood-plastic composite (WPC) is manufactured using waste wood and plastics, and its use is increasing in outdoor furniture making among various other uses of WPC. However, it is difficult to biodegrade WPC, and WPC may cause various environmental problems when it is simply incinerated or filled on land, as WPC contains a difficult-to-decompose polymer. Recently, Kim et al. [9] developed a technology for producing aromatic compounds of high utility value as fuels and chemical raw materials through pyrolysis and catalytic thermal decomposition of WPC.

In addition, it was reported by Ghorbanzhad et al. [10] that the synergistic formation of aromatic compounds from the interaction of the respective plastic and biomass pyrolytic reaction intermediates (olefins

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from plastic pyrolysis and furans from biomass pyrolysis) could be achieved by co-feeding plastics and biomass in CFP. Hence, CFP of WPC can be a useful recycling technology enabling the production of aromatic hydrocarbons because WPC consists of biomass, PE, PP, and inorganic additives.

Meanwhile, the efficient commercialization of CFP of biomass or plastics is still challenged due to the burden on the CFP caused by the high catalytic cost. Recently, Ro et al. [11] reported the possibility of reducing the CFP cost while ensuring a catalytic effect above a certain acceptable level by using natural catalysts. Shim et al. [12] also found the catalytic effect of low-cost natural zeolite on converting oxygenates to aromatic hydrocarbons via the catalytic pyrolysis of chicken manure. Aisien et al. [13] indicated that spent fluid catalytic cracking (FCC) catalyst can provide the higher cracking efficiency and increase the production of gasoline and diesel oil on the catalytic pyrolysis of polypropylene. However, although low-cost/natural catalysts have the potential to be effective and economical in the production of aromatic compounds from different feedstocks and under different techniques, research on low-cost catalysts for a WPC feedstock has not yet been reported.

So, in this study, γ -Al₂O₃, spent FCC catalyst, and natural zeolite were used in the CFP of WPC (for aromatics production) as low-cost catalysts to reduce the CFP cost. In particular, the performance of these catalysts was evaluated using a thermogravimetric (TG) analyzer in terms of the thermal decomposition temperature change and activation energy distribution of the CFP. In addition, a pyrolyzer-gas chromatography/mass spectrometry (Py-GC/MS) analysis was performed to evaluate and compare the change in the thermal decomposition products and the aromatic compound production efficiency of the CFP under the different catalysts.

Experiments

Wood-plastic composite

Powder forms of WPC (with particle size < 250 μ m), manufactured with woody components (58 %), PE (9 %), PP (18 %), CaCO₃ (8.5 %), and other additives (6.5 %), were purchased from companies in South Korea. The WPC was then dried at 70 °C for a day. Table 1 shows the proximate and ultimate analysis results of WPC. Subsequent proximate analysis of the WPC showed that the WPC had a high volatiles content (85.6 %) and a small amount of fixed carbon (5.0 %), ash (7.6 %), and water (1.8 %), respectively. In addition, the carbon, oxygen, hydrogen, and nitrogen content of the WPC was 54.0, 37.1, 8.5, and 0.4 %, respectively, suggesting the potential use of WPC as fuel or chemical feedstock.

Catalysts characterization

The BET surface area (S_{BET}) and pore size of each catalyst were obtained from the BET analysis according to a process described in the

Table 1
Proximate and ultimate analysis result of WPC.

wt %	WPC	
Proximate analysis	Water	1.8
	Volatiles	85.6
	Fixed carbon	5.0
	Ash	7.6
	Sum	100
Ultimate analysis (Dry ash free basis)	C	54.0
	O*	37.1
	H	8.5
	N	0.4
	Sum	100

* By difference.

literature [14]. The BET analysis findings are shown in Table 2. Among the catalysts, spent FCC catalyst had the largest pore size (5.2 nm) due to the binding material [15], followed by that of γ -Al₂O₃ (3.0 nm) and natural zeolite (0.5 nm), respectively.

Subsequently, the temperature-programmed desorption of ammonia (NH₃-TPD) analysis of the catalysts was performed by referring to a process reported in the literature [16]. Briefly, the catalysts were pre-heated at 500 °C for 30 min to eliminate their impurities and cooled down to 100 °C to absorb NH₃. After the NH₃ absorption, the physically absorbed NH₃ of the catalysts was eliminated at 100 °C under 30 ml/min of helium flow, and the catalysts were then heated to 650 °C for acidity analysis. Further, the amount of desorbed NH₃ from the catalysts was monitored using a thermal conductivity detector. Eventually, the NH₃-TPD spectra of the catalysts, shown in Fig. 1, had two peaks (at low- and high-temperature regions, respectively), suggesting the presence of weak and strong acidic sites in the catalysts [17]. In particular, natural zeolite had the largest acidity, followed by that of γ -Al₂O₃ and spent FCC catalyst, respectively. Also, the NH₃-TPD spectrum of natural zeolite had a strong peak at a temperature higher than 300 °C, unlike the other catalysts, suggesting the strong acidity of natural zeolite.

TGA

WPC (1 mg) and a mixture of WPC/catalyst were heated from ambient temperature to 800 °C under 50 ml/min of nitrogen flow at varied heating rates of 10, 20, and 30 °C/min in a TG analyzer (Pyris 1, Perkin-Elmer) for the analysis. Subsequently, a kinetic analysis was performed on the TGA results using the revised Ozawa method [18] to find the apparent activation energy (E_a) of the non-catalytic (NC) and CFP of WPC.

Py-GC/MS analysis

Py-GC/MS analysis of NC and CFP of WPC was performed to determine their product distribution. Particularly, for this analysis, 0.5 mg of WPC was decomposed in a micro-pyrolyzer (PY-3030D, Frontier Laboratories) at 600 °C under a catalyst/no catalyst, and the pyrolytic chemical products were analyzed using GC/MS7890A/5975C, Agilent Technology gas chromatograph (GC). Briefly, the pyrolytic vapor was sent to the GC inlet (kept at 320 °C and had a split ratio of 100/1) and subsequently introduced into the capillary column (UA-5, 30 m length \times 0.25 mm inner diameter \times 0.25 μ m film thickness) installed in the GC. Next, the temperature of the GC oven was programmed to be varying from 40 °C (for 5 min) to 320 °C (for 15 min) at the rate of 15 °C/min. Finally, the pyrolytic chemical products were identified by comparing the mass spectrum of each peak on the total ion chromatogram (TIC) with that in libraries (NIST08th version, F-Search library). Notably, for the case of the CFP, the same amount of catalyst (0.5 mg) and WPC (0.5 mg) were mixed for 1/1 of catalyst/WPC. Additionally, 5/1 of catalyst/WPC (with 2.5 mg of catalyst and 0.5 mg of WPC) was also prepared for analysis to study the effect of catalytic loading on aromatics production.

Result and discussion

Kinetic analysis

The decomposition temperature region of WPC can be divided into

Table 2
BET surface area and pore size of natural zeolite, spent FCC catalyst, and γ -Al₂O₃.

Catalyst	S_{BET} (m ² /g)	Pore size (nm)
Natural zeolite	157	0.5
Spent FCC catalyst	165	5.2
γ -Al ₂ O ₃	70	3.0

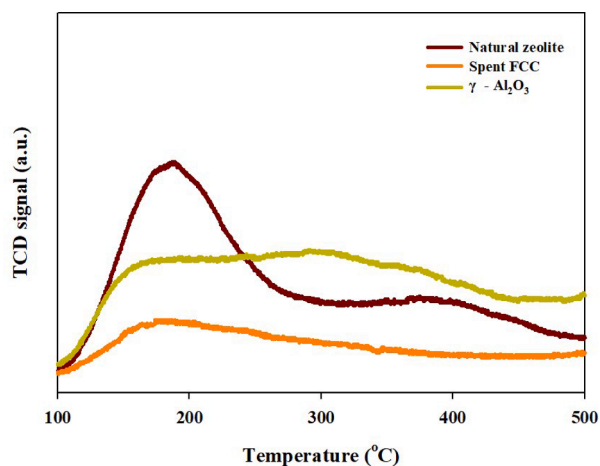
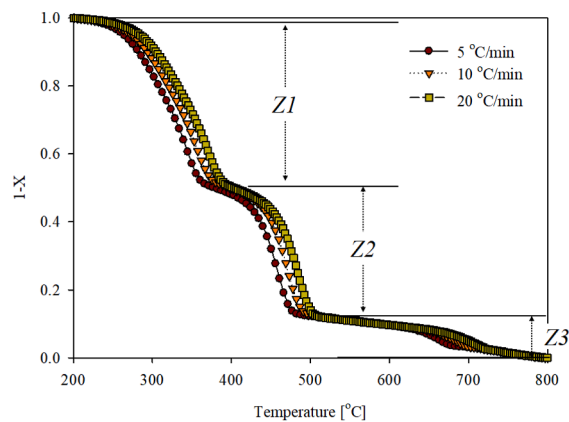


Fig. 1. NH_3 -TPD spectra of natural zeolite, spent FCC catalyst, and $\gamma\text{-Al}_2\text{O}_3$.

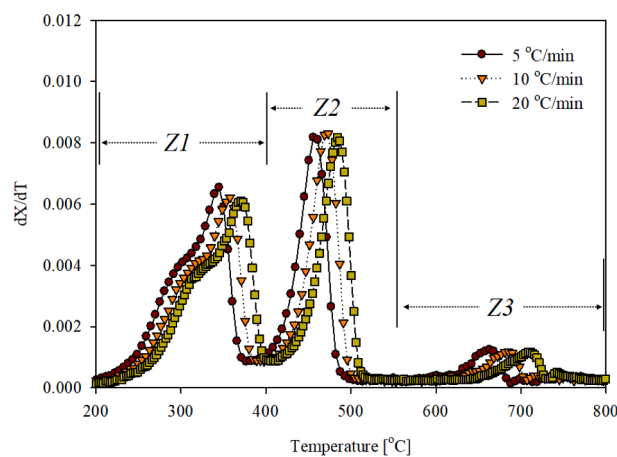
the decomposition of biomass at 200–400 °C (Z1) [19], synthetic plastics (PE and PP, [20]) at up to 500 °C (Z2), and the emission of CO_2 from CaCO_3 at temperatures higher than 600 °C (Z3) [21]. Although natural zeolite has a strong acidity and small pores, the decomposition pattern and temperature regions for the decomposition of biomass and CaCO_3 (Fig. 3) were not much different from those of the NC pyrolysis of WPC reported in our previous publication (Fig. 2. [22]). However, the DTG peak height for the decomposition of plastics decreased, and the peak was broader, suggesting a good catalytic effect of natural zeolite in the cracking of PP and PE in WPC. In addition, the maximum decomposition temperature of plastic (Z2) on the DTG curve of CFP of WPC under natural zeolite, 450 °C, was also lower than that on the DTG curve of NC pyrolysis of WPC, 470 °C, confirming that natural zeolite had a good catalytic effect in the cracking of PP and PE [14]. As seen on Table 2 and Fig. 1, the pore size of natural zeolite is small (0.5 nm), however, its acidity is higher than other catalysts used in this study. This suggests that the catalytic effect to plastics (PE and PP) is more effective than that to biomass because the small pore size of catalyst can limit the diffusion of large molecular biomass pyrolyzates.

Fig. 4 shows the apparent activation energies of NC and CFP of WPC calculated from the kinetic analysis based on the Ozawa method. In addition, the linearity of the plots of $\ln\beta$ versus $1/T$ at different fractions of WPC conversion in the NC and CFP of WPC was also high (with $R^2 > 0.96$), suggesting the accuracy of the kinetic analysis of this study (Fig. S1).

Notably, the large pore size and S_{BET} of spent FCC catalyst could provide an easy migration of reactant molecules during the CFP of WPC over spent FCC catalyst, leading to accelerated decomposition of WPC, according to Fig. 4. Likewise, the E_a at the initial stage of the WPC decomposition over natural zeolite and $\gamma\text{-Al}_2\text{O}_3$, respectively, was higher than that over spent FCC catalyst because of the low migration of biomass reactant molecules caused by the smaller pore sizes of natural zeolite and $\gamma\text{-Al}_2\text{O}_3$. However, the E_a towards the end of the WPC conversion over natural zeolite and $\gamma\text{-Al}_2\text{O}_3$, respectively, largely decreased with the increase in the fraction of WPC conversion, suggesting the effectiveness of natural zeolite and $\gamma\text{-Al}_2\text{O}_3$ in the decomposition of PE and PP due to the high acidity of natural zeolite and $\gamma\text{-Al}_2\text{O}_3$. Among the different WPC decompositions, the average E_a of the WPC conversion over natural zeolite (127.5 kJ/mol) was the lowest, followed by that over spent FCC catalyst (133.3 kJ/mol) and $\gamma\text{-Al}_2\text{O}_3$ (145.0 kJ/mol), respectively, suggesting the high efficiency of natural zeolite in the CFP of WPC. In addition, as shown in Fig. 5, the much larger decrease of T_{max} in Z2 of the decomposition of WPC over natural zeolite and spent FCC catalyst, respectively, compared to the NC and CFP over $\gamma\text{-Al}_2\text{O}_3$ of WPC, as well confirmed the high catalytic effect of natural zeolite. Finally, although high-temperature pyrolysis could facilitate high WPC cracking



(a) TG curves



(b) DTG curves

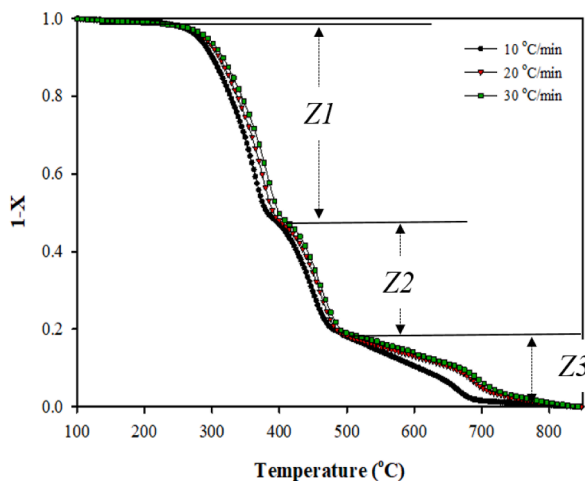
Fig. 2. (a) TG and (b) DTG curves of non-catalytic pyrolysis of WPC at varied heating rates of 5, 10, and 20 °C/min [22].

efficiency, the decomposition of CaCO_3 in WPC pyrolysis at temperatures higher than 600 °C suggested a large amount of CO_2 formation via the pyrolysis of WPC. Therefore, 600 °C was set as the reaction temperature for the Py-GC/MS analysis of NC and CFP of WPC.

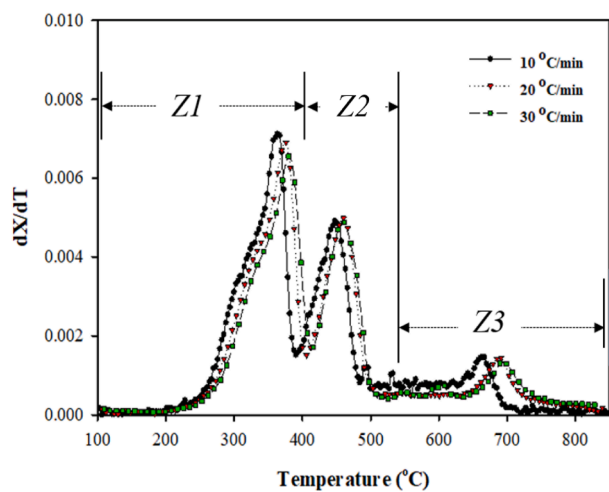
Py-GC/MS analysis

Pyrolysis of WPC produced hydrocarbons, such as linear and iso-hydrocarbons, and oxygen-containing pyrolyzates from the decomposition of biomass, PE, and PP in WPC, as shown in Fig. 6 and Table 3. As expected, the NC pyrolysis of WPC produced large amounts of oxygenates and hydrocarbons mainly. Likewise, the pyrolysis of wood powder in WPC produced oxygen-containing compounds (levoglucosan, furans, acids, aldehydes, ketones, alcohols, and phenolics) due to the decomposition of biomass components [23] and that of PE and PP in WPC generated linear and branched hydrocarbons (paraffin and olefin) having a wide carbon number distribution containing wax to cause the low quality of this pyrolysis product [24].

Furthermore, the amounts of produced oxygenates, linear, and branched hydrocarbons of the pyrolysis decreased with the use of the catalysts, with a corresponding increase in the amount of aromatic hydrocarbons produced, as shown in Fig. S2 [25]. Compared to NC pyrolysis of WPC, wax compounds and oxygenates of WPC was reduced via



(a) TG curves



(b) DTG curves

Fig. 3. (a) TG and (b) DTG curves of catalytic pyrolysis of WPC over natural zeolite at varied heating rates of 10, 20, and 30 °C/min.

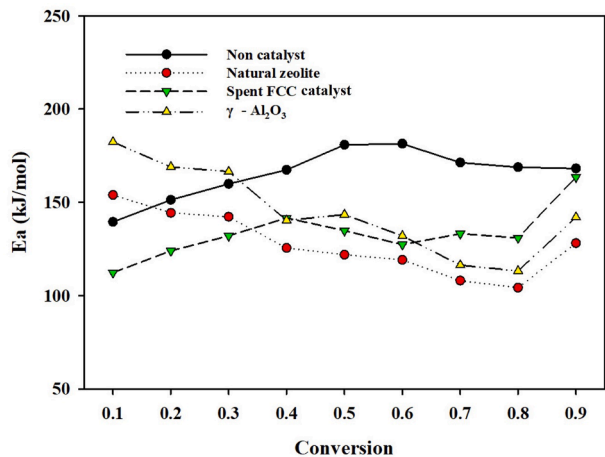
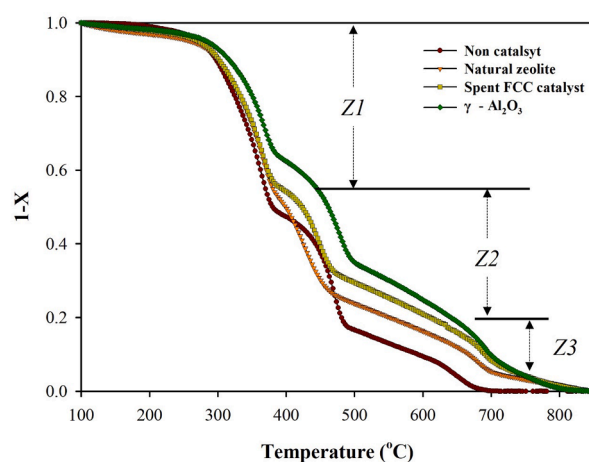
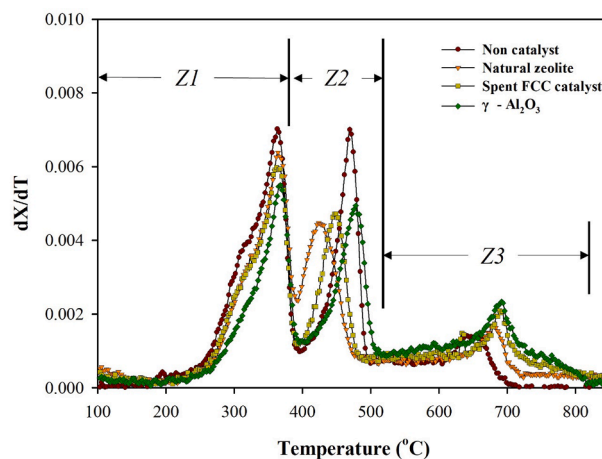


Fig. 4. Apparent activation energies of non-catalytic and catalytic pyrolysis of WPC from the kinetic analysis based on the Ozawa method.



(a) TG curves



(b) DTG curves

Fig. 5. (a) TG and (b) DTG curves of catalytic pyrolysis of WPC over low-cost catalysts of this study with a catalyst/WPC of 5/1.

the CFP over spent FCC catalyst and $\gamma\text{-Al}_2\text{O}_3$, because these catalysts have large pore size that can provide the easier diffusion of large molecules. However, spent FCC catalyst and $\gamma\text{-Al}_2\text{O}_3$ could not significantly increase production of aromatic hydrocarbons due to their low acidity. On the other hand, high-acidity active sites of natural zeolite decomposed high molecular hydrocarbons into $\text{C}_3 \sim \text{C}_5$ hydrocarbons efficiently and converted them into aromatics through a cyclization reaction, leading the increased formation of aromatic hydrocarbons. The larger amount of aromatic hydrocarbons was achieved by increasing catalyst/feedstock ratio from 1/1 to 5/1 with the further decrease of oxygenates, suggesting the increased catalytic effect due to the larger amount of catalyst use.

Conclusion

The catalytic pyrolysis of WPC over low-cost catalysts, natural zeolite, spent FCC catalyst, and $\gamma\text{-Al}_2\text{O}_3$, was investigated in this study to decrease the overall cost of aromatics production from this pyrolysis. Although the use of low-cost catalysts could not affect the decomposition temperature of wood powder, they effectively accelerated the decomposition of PE and PP in WPC with a corresponding decrease in the apparent activation energy of the pyrolysis. Among the low-cost catalysts of this study, natural zeolite performed the best in the WPC decomposition and also increased the formed amount of aromatic hydrocarbons at 600 °C, followed by that over spent FCC catalyst and

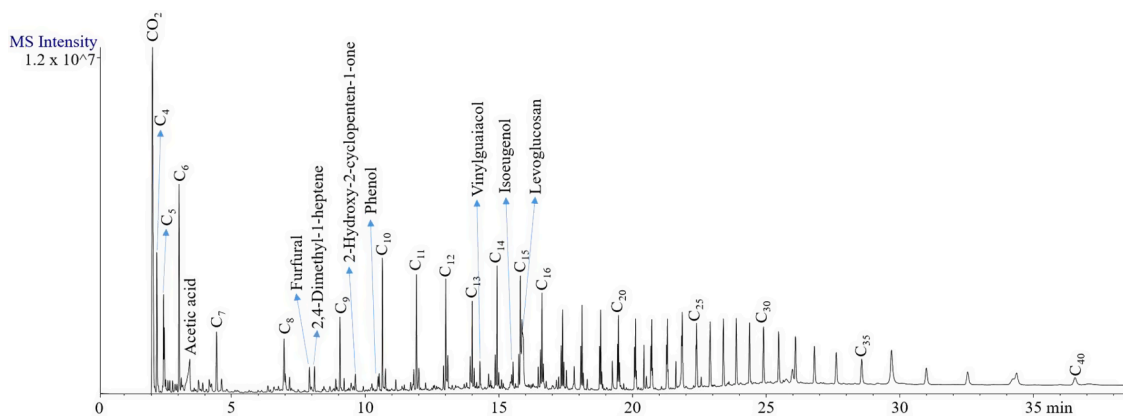


Fig. 6. Product distribution of non-catalytic pyrolysis of WPC.

Table 3

MS peak areas for the products of non-catalytic and catalytic pyrolysis of WPC over the low-cost catalysts of this study.

MS peak area $\times 10^6$	Non-catalytic pyrolysis of WPC	Catalyst/WPC: 1/1			Catalyst/WPC: 5/1		
		Natural zeolite	Spent FCC catalyst	γ -Al ₂ O ₃	Natural zeolite	Spent FCC catalyst	γ -Al ₂ O ₃
Acids	83.5	29.3	35.5	46.4	26.4	35.9	35.3
Aldehydes	16.1	7.6	Not determined (N.D.)	N.D.	N.D.	N.D.	N.D.
Ketones	28.4	15.2	14.9	12.6	3.1	12.9	12.7
Furans	54.3	113.6	80.3	62.1	93.4	105.1	69.4
Alcohols	N.D.	N.D.	5.1	10.2	N.D.	N.D.	9.1
Phenolics	81.7	54.5	84.7	50.4	37.8	60.4	6.2
Oxygenates	264.0	220.2	220.5	181.7	160.7	214.3	132.7
BTEXs	5.7	77.9	19.4	11.4	472.3	55.1	27.7
Other MAHs	N.D.	28.6	N.D.	N.D.	79.3	51.3	N.D.
Naphthalenes	N.D.	22.8	N.D.	N.D.	87.2	30.6	N.D.
Aromatics	5.7	128.3	19.4	11.4	638.8	137	27.7
Paraffins	542.5	150.8	186.6	197.2	3.2	106.9	300.6
Olefins	1,295.3	871	763.8	744.9	1158.2	638.6	797.2
Naphthenes	40.4	129	79.1	55.3	62.3	120.9	34.1
Other hydrocarbons	1878.1	1150.8	1029.5	997.4	1223.7	866.4	1131.9
N-containing compounds	29.3	6.1	15.3	13	N.D.	5.5	15.8

γ -Al₂O₃, respectively, due to its high acidity and small pore size. In addition, increasing the catalytic loading in the CFP of WPC increased the production of aromatic hydrocarbons and could estimate the potential use of low-cost catalysts in WPC pyrolysis.

CRedit authorship contribution statement

Hyeon Su Heo: Writing – original draft. Sumin Pyo: Writing – original draft. Bo Sung Kang: Writing – original draft. Jung Sul Jung: Writing – original draft. Gwang Hoon Rhee: Writing – review & editing, Writing – original draft. Young-Min Kim: Writing – review & editing, Writing – original draft. Ji Man Kim: Writing – review & editing, Writing – original draft. Young-Kwon Park: Writing – review & editing, Writing – original draft.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

The authors do not have permission to share data.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at <https://doi.org/10.1016/j.seta.2023.103051>.

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