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Synthesis of Fe- and Ce- Incorporated Extracted Aluminium (EA) Catalysts to Produce Bio-Oil via Catalytic Co-Pyrolysis of Biomass and Plastic

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ABSTRACT

Pyrolysis is a promising route for converting biomass and plastic waste into valuable biofuels such as bio-oil. However, bio-oil derived from biomass typically has poor quality due to its high oxygenate compound content. Catalytic co-pyrolysis of biomass and plastics offers an effective strategy for enhancing bio-oil quality by promoting deoxygenation. In addition, the use of sludge-derived catalysts offers a sustainable approach to valorizing industrial waste while reducing reliance on commercial catalyst supports. This study investigates the effect of metal types in sludge-derived metal oxide catalysts on bio-oil production from the catalytic co-pyrolysis of cotton fabric and polypropylene wastes. Iron (Fe) and cerium (Ce) (10 wt.%) were incorporated into extracted aluminum (EA) using the wet impregnation method. Catalyst properties were characterized using BET and SEM, while the chemical component distribution of bio-oil was analyzed using GC-MS. The results show that 10Fe–EA produced a higher bio-oil yield (33.3 wt.%) than 10Ce–EA (27.2 wt.%). This performance is attributed to its higher surface area and pore volume. GC–MS analysis quantified that 10Fe–EA promoted the formation of hydrocarbons, predominantly cycloalkanes and alkenes, while suppressing oxygenated compounds. These findings demonstrate the potential of waste-derived Fe-supported extracted aluminum as an effective catalyst for upgrading bio-oil during catalytic co-pyrolysis, offering a sustainable pathway for the simultaneous valorization of industrial sludge and the production of renewable fuel.

Keywords: Catalytic co-pyrolysis; Biomass; Plastic; Metal oxide; Sludge; Bio-oil.

1. INTRODUCTION

The increasing generation of solid waste and the growing demand for sustainable energy have become major global challenges. Rapid industrialization, urbanization, and population growth have significantly increased the production of biomass and plastic wastes, leading to environmental pollution and pressure on landfill capacity. At the same time, the depletion of fossil fuel resources and concerns over greenhouse gas emissions have intensified the search for renewable and environmentally friendly energy alternatives. Converting waste materials into valuable energy products offers a promising approach to simultaneously address waste management and energy security issues [1, 2].

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Among the available waste-to-energy technologies, pyrolysis has gained significant attention due to its ability to convert biomass and plastic waste into liquid fuels, gases, and char under oxygen-limited conditions. However, biomass pyrolysis is constrained by high moisture and ash contents, low calorific value, heterogeneous composition, and supply-chain challenges [3], whereas plastic pyrolysis is often hindered by poor heat transfer, high melt viscosity, and reactor clogging [4]. To overcome these limitations, the co-pyrolysis of biomass and plastics has emerged as a promising approach that leverages synergistic interactions between feedstocks, thereby improving product yield and quality [5]. Moreover, pyrolysis is recognized as an important chemical recycling technology for large-scale plastic waste management and contributes to several Sustainable Development Goals (SDGs), including affordable and clean energy (SDG 7) and climate action (SDG 13) [6].

In addition, catalytic pyrolysis has gained attention as an effective technique to upgrade bio-oil. Therefore, catalyst development has become an important research area to enhance process efficiency and improve bio-oil quality [7, 8]. The integration of metal oxide catalysts during catalytic co-pyrolysis can significantly improve process efficiency and bio-oil quality by promoting reactions such as cracking, deoxygenation, and decarboxylation, thereby reducing oxygenated compounds and increasing fuel quality [9]. Metal oxides such as Fe_2O_3 and CeO_2 are particularly effective in facilitating O_2 transfer and hydrogenation [10]. Meanwhile, Al_2O_3 provides a suitable support with well-distributed acidic sites. Consequently, metal oxide catalysts play a vital role in enhancing product quality, thermal stability, and overall catalytic co-pyrolysis performance [11].

To address the sustainability issues associated with conventional catalysts, increasing attention has been directed towards waste-derived materials as alternative catalyst supports and active catalysts. Several studies have reported the utilization of waste-derived materials as active catalysts and catalyst supports, such as red mud [12], spent fluid catalytic cracking (FCC) catalyst [13], electric arc furnace slag [14], and biochar [15]. The use of waste-derived materials as catalysts offers a sustainable strategy to minimize waste disposal while promoting resource recovery and advancing circular economy initiatives. Among these materials, industrial sludge has attracted considerable attention as a valuable secondary resource due to its high content of recoverable metal oxides and minerals suitable for catalyst synthesis. In particular, aluminum is one of the most abundant and valuable metals present in industrial sludge and can be recovered and converted into aluminum oxide (Al_2O_3), a widely used catalyst support in heterogeneous catalysis. The widespread use of Al_2O_3 is attributed to its desirable physicochemical properties, including a high specific surface area, excellent thermal stability, mechanical strength, and strong metal–support interactions that facilitate the dispersion of active metal species. These characteristics enhance the accessibility and stability of catalytically active sites, making Al_2O_3 an effective support for various catalytic reactions [16]. These physicochemical characteristics enable Al_2O_3 to function effectively as both an active catalytic phase and a robust catalyst support, making it an attractive material for heterogeneous catalytic applications.

Recovering aluminum from sludge not only reduces the volume of waste sent to landfills and the associated risks of soil and groundwater contamination [17]. Consequently, aluminum recovery contributes to resource conservation, waste minimization, and the transition toward a circular economy by transforming industrial waste into value-added materials [18]. Furthermore, several studies have demonstrated that aluminum recovered from sludge can be effectively utilized as a catalyst in various applications, including microbial fuel cells [19], antibiotic degradation [20], and hydrogen production [21]. In these applications, the recovered aluminum-based catalysts exhibited promising catalytic performance, thereby enhancing reaction efficiency. The transition metal oxides, particularly Fe and Ce, have attracted considerable attention because of their excellent redox properties, oxygen mobility, and ability to promote cracking and deoxygenation reactions during catalytic pyrolysis. Although catalytic co-pyrolysis has shown significant potential to improve bio-oil quality, the selection of sustainable catalysts remains a major

challenge. These findings demonstrate the feasibility of converting aluminum-rich sludge into high-value catalyst materials. However, the application of sludge-derived aluminum as a catalyst support for biomass–plastic catalytic co-pyrolysis remains largely unexplored.

Although considerable progress has been made in developing waste-derived catalysts, the utilization of aluminum recovered from industrial sludge as a catalyst support for catalytic co-pyrolysis has received limited attention. Moreover, the effects of Fe and Ce incorporation on the physicochemical properties of extracted aluminum, as well as on bio-oil production and hydrocarbon component distribution, remain poorly understood. It is hypothesized that the combination of sludge-derived Al_2O_3 with Fe and Ce active phases will produce a highly dispersed catalyst with enhanced cracking and deoxygenation capabilities, leading to improved bio-oil quality. Accordingly, this study synthesizes Fe- and Ce-modified catalysts extracted from industrial sludge, characterizes their physicochemical properties, and evaluates their catalytic activity in the co-pyrolysis of cotton fabric waste (CFW) and polypropylene waste (PPW).

2. MATERIAL AND METHODS

2.1 Materials

Industrial sludge was utilized as the source of aluminum for catalyst synthesis. Prior to aluminum extraction, the sludge was dried at 110 °C for 6 h, ground into a fine powder, and sieved to obtain particles smaller than 250 μm. The resulting extracted aluminum (EA) was subsequently used as a catalyst support and impregnated with iron and cerium precursors. Iron nitrate nonahydrate ($Fe(NO_3)_3{\cdot}9H_2O$) at 98% purity from QreC and cerium nitrate hexahydrate ($Ce(NO_3)_3{\cdot}6H_2O$) at 98% purity from QreC were employed as the Fe and Ce sources, respectively, for the synthesis of Fe-EA and Ce-EA catalysts. Cotton fabric waste (CFW) and polypropylene plastic waste (PPW) were selected as feedstocks for the catalytic co-pyrolysis experiments. Prior to use, both materials were cut into approximately 2 mm pieces to ensure homogeneous mixing and efficient heat transfer during the pyrolysis process [22].

2.2 Catalyst synthesis and characterization

The catalyst synthesis was carried out in two stages. In the first stage, aluminum was extracted from the industrial sludge, followed by the incorporation of the desired metal into the extracted aluminum in the second stage. The synthesis procedure was adapted and modified from the method reported by Bashah et al. [22].

2.2.1 Preparation of extracted aluminum

50 g of dried sludge was mixed with 200 mL of 2 M H_2SO_4 in a covered beaker and subjected to acid leaching at 70 °C under continuous stirring at 400 rpm for 30 min. Upon completion of the leaching process, the slurry was cooled to room temperature and allowed to settle overnight to facilitate solid-liquid separation. The suspension was subsequently centrifuged at 3000 rpm for 5 min, and the supernatant was recovered by vacuum filtration to obtain a clear aluminum-rich leachate. The aluminum-rich solution was then precipitated by dropwise addition of aqueous ammonia to a final pH of 9. The resulting precipitate was collected via vacuum filtration and repeatedly washed with deionized water until a neutral pH was attained to remove residual ammonia and soluble impurities. The washed precipitate was dried in an oven (Model Mermet) at 110 °C for 12 h and subsequently calcined in a muffle furnace (model Carbolite) at 600 °C for 2 h with a heating rate of 5 °C·min^{-1}. Subsequently, the calcined EA was ground to a fine powder using a mortar and pestle and was ready to be impregnated with Fe and Ce.

2.2.2 Synthesis of catalysts

A catalyst containing 10 wt.% Fe was initially prepared by dissolving the required amount of iron nitrate nonahydrate ($Fe(NO_3)_3 \cdot 9H_2O$) in deionized water, followed by the addition of 3 g of EA support. The resulting slurry was stirred at room temperature at 250 rpm for 4 h to promote uniform dispersion of the metal precursor onto the support surface. The impregnated sample was subsequently dried in an oven (Model Mermet) at 115 °C for 16 h, ground into a fine powder, and calcined in air at 600 °C for 4 h with a heating rate of 5 °C·min^{-1} using a muffle furnace (model Carbolite). The resulting catalyst was designated as 10Fe-EA, indicating 10 wt.% Fe loading on the EA support. A similar procedure was employed for the synthesis of the Ce-modified catalyst using cerium nitrate hexahydrate ($Ce(NO_3)_3 \cdot 6H_2O$) as the precursor. The resulting catalyst was denoted as 10Ce-EA, corresponding to a 10 wt.% Ce loading on the EA support. Subsequently, the catalytic activity of the synthesized 10Fe-EA and 10Ce-EA catalysts was evaluated in the catalytic co-pyrolysis of cotton fabric waste (CFW) and polypropylene waste (PPW).

2.2.3 Catalyst characterization

The physicochemical properties, such as surface morphology and textural properties, of the synthesized 10Fe-EA and 10Ce-EA catalysts were characterized using scanning electron microscopy (Hitachi Regulus 8220) and Nitrogen sorption-desorption (Micromeritics, ASAP 2020), respectively. The catalyst exhibited the best catalytic activity, as evidenced by the highest bio-oil yield and the highest degree of deoxygenation.

2.3 Catalytic Co-pyrolysis of CFW and PPW

Catalytic co-pyrolysis experiments were conducted in a fixed-bed reactor (Figure 1) at 600 °C for 30 min under a continuous nitrogen flow of 120 mL·min^{-1}. These conditions were selected to achieve near-complete thermal decomposition of the feedstock while minimizing excessive secondary cracking of the pyrolysis vapors.

Figure 1: Reactor system for catalytic co-pyrolysis of cotton fabric waste and polypropylene plastic waste.

Prior to each experiment, the reactor was purged with nitrogen to establish an inert atmosphere and eliminate residual air. A mixture consisting of 0.5 g cotton fabric waste (CFW), 0.5 g polypropylene waste (PPW), and 1 g catalyst was loaded into the reactor, with quartz wool used to support and separate the catalyst and feedstock layers. At the end of the reaction, the condensable products were collected from the wax trap and condenser. The combined liquid

fraction was designated as bio-oil and subsequently subjected to further characterization using a gas chromatography-mass spectrometry (GC-MS) model QP2010 Ultra (Shimadzu). The component distribution of bio-oil was performed using a BPX5 capillary column (29.5 m × 0.25 mm × 0.25 mm). High-purity helium (99.99%) was used as the carrier gas at a flow rate of 1.0 $mL \cdot min^{-1}$. The product yield (char, bio-oil and gas) and selectivity were calculated by equations (1)-(3) adapted from Bashah et al. [23], while the degree of deoxygenation was calculated by equation (4) adapted from Kaewpengkrow et al. [24].

Char and bio-oil yield, % = (weight of product/weight of feedstock) x 100 (1)

Gas yield, % = 100 – yield of char – yield of bio-oil (2)

Bio-oil component distribution, % = (3)
(sum area of compound/ total area of all compounds) x 100

Degree of deoxygenation, %DOD = (4)
1-(% oxygenated in catalytic/% oxygenated without catalyst) x 100

3. RESULTS AND DISCUSSION

3.1 Catalytic co-pyrolysis of CFW and PPW

The Ce-EA and Fe-EA catalysts were prepared and used in the co-pyrolysis of CFW and PPW. The products from co-pyrolysis, which consist of char, bio-oil, and gas, and their bio-oil component distribution, were evaluated and compared without a catalyst and with the 10Ce-EA and 10Fe-EA catalysts. Furthermore, the effect of metal loadings on product yield and the distribution of bio-oil components is investigated. The selection of the best metal and metal loadings is based on the highest bio-oil yield. Figure 2 shows the product yield of co-pyrolysis of CFW and PPW without a catalyst and with 10Fe-EA and 10Ce-EA catalysts.

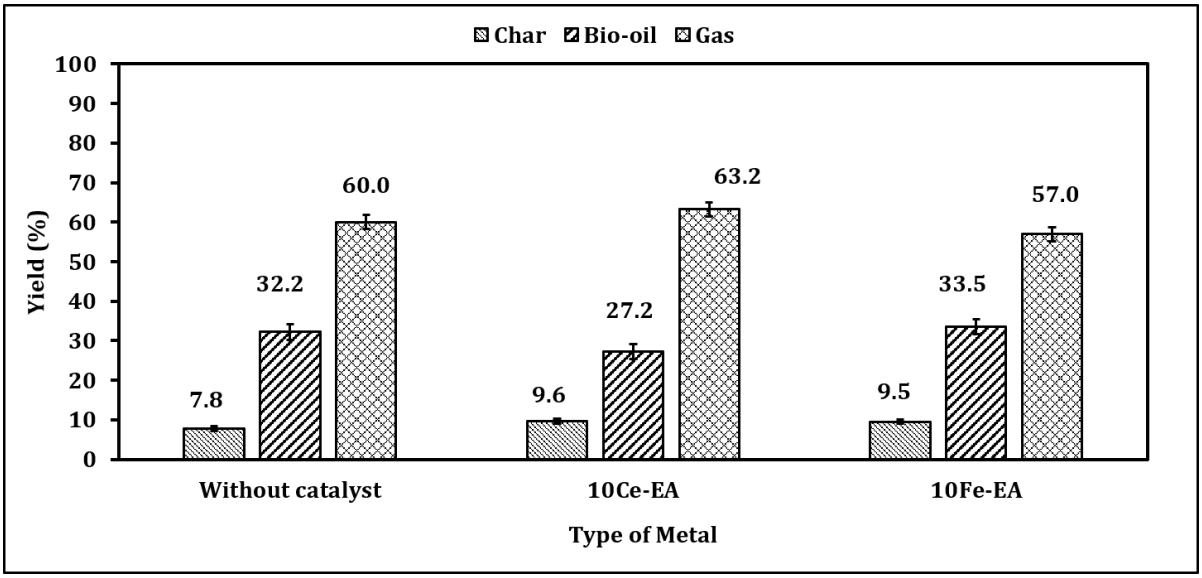

Figure 2: Product yield without catalyst and catalyst in co-pyrolysis of CFW and PPW (Operating Condition: 600 °C Pyrolysis Temperature; 60 min Pyrolysis Time; 50:50 CFW/PPW Ratio; 1:1 F/C Ratio).

The result shows that the yield of char produced is 7.8% for co-pyrolysis without catalysts (CPWC), slightly lower than those for 10Ce-EA and 10Fe-EA, which are 9.6% and 9.5%, respectively. However, the slight differences in char yield between catalysts and CPWC were

relatively small, suggesting that the ex-situ catalyst had a negligible influence on primary char formation, as the catalyst was not in direct contact with the feedstock during the thermal degradation period [1]. The 10Fe–EA catalyst produced a bio-oil yield of 33.5 wt.%, which was slightly higher than that obtained from non-catalytic co-pyrolysis (32.2 wt.%) and the 10Ce–EA catalyst (27.2 wt.%). In contrast, the 10Ce–EA catalyst generated the highest gas yield (63.2 wt.%), followed by non-catalytic co-pyrolysis (60.0 wt.%) and the 10Fe–EA catalyst (57.3 wt.%). The catalytic co-pyrolysis of CFW and PPW showed that the addition of the catalyst caused only a slight change in bio-oil yield compared with the non-catalytic process. This suggests that the catalyst does not primarily contribute to increasing the quantity of bio-oil produced. However, it facilitates secondary vapor-phase reactions, including cracking and deoxygenation, which modify the composition and enhance the quality of the resulting bio-oil. Similarly, Lui et al. [25] found that the Fe/Al_2O_3 and Ce/Al_2O_3 catalysts increased deoxygenation activity in corncob pyrolysis, thereby improving hydrocarbon composition by reducing oxygenate compounds. The catalytic activity was strongly influenced by the physicochemical characteristics of the prepared catalysts, particularly their textural properties and surface morphology.

Table 1 shows the textural properties (surface area and pores) of 10Ce-EA and 10Fe-EA. It is observed that 10Fe-EA possesses a substantially larger surface area (75.205 $m^2 \cdot g^{-1}$) and pore volume (0.0981 $cm^3 \cdot g^{-1}$) compared with the 10Ce–EA catalyst (1.327 $m^2 \cdot g^{-1}$ and 0.0042 $cm^3 \cdot g^{-1}$, respectively).

Table 1: Surface area and pores analysis using N_2 adsorption and desorption.

Catalyst	10Ce-EA	10Fe-EA
Surface area (m^2/g)	1.327	75.205
Pore volume (cm^3/g)	0.0042	0.0981
Pore size (nm)	12.551	5.217

The addition of Ce into the EA support surface leads to a lower surface area and pore volume. This finding indicates the diffusion of metals on the support surface. The distribution of Ce on the EA support has led to partial pore blockage, thereby reducing the surface area [26]. A similar reduction in textural properties following Ce loading has been reported for ceria-supported catalysts and is commonly associated with partial pore filling, surface coverage, and crystallite growth of CeO_2 [27-28].

3.2 Morphology of Ce-EA and Fe-EA catalysts

The SEM image in Figure 3 further confirms the surface morphology of the catalysts.

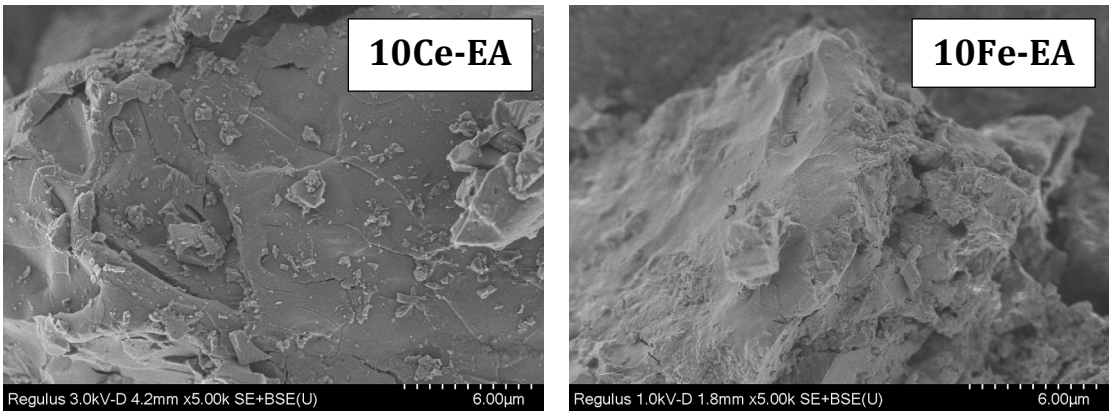

Figure 3: Catalyst surface morphology of 10Ce-EA and 10Fe-EA catalysts at 5000× magnification.

The 10Ce-EA catalyst exhibited a relatively dense and compact surface, whereas the 10Fe-EA catalyst displayed a rough, irregular, and fractured morphology with visible cavities. The rougher morphology of the 10Fe-EA catalyst indicates a less compact surface compared to 10Ce-EA. These morphological observations are consistent with the nitrogen adsorption-desorption analysis, which showed that 10Fe-EA possessed a higher specific surface area than 10Ce-EA. The higher surface area and improved pore characteristics are expected to enhance the diffusion of pyrolysis vapors and increase the availability of accessible catalytic sites, thereby promoting more effective vapor–catalyst interactions.

3.3 Bio-oil component distribution

Figure 4 depicts the comparison of chemical compound distribution in bio-oil for 10Ce-EA, 10Fe-EA and without a catalyst, and the degree of deoxygenation (DOD) of bio-oil for each catalyst.

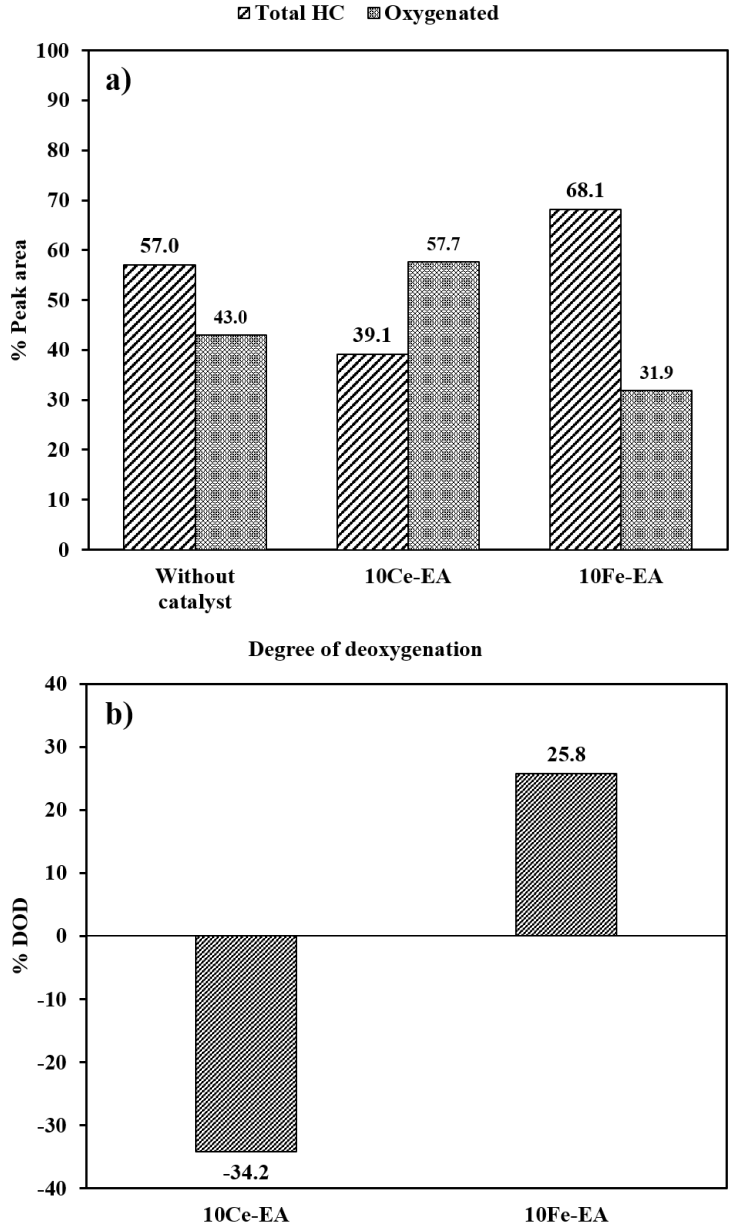

Figure 4: Chemical component distribution (a) Hydrocarbons and oxygenated compound selectivity (b) Degree of deoxygenation of 10Ce-EA, 10Fe-EA and without catalyst (Operating Condition: 600 °C Pyrolysis Temperature; 60 min Pyrolysis Time; 50:50 CFW/PPW Ratio; 1:1 F/C Ratio).

The finding shows that 10Fe-EA produced the highest total hydrocarbon yield (68.1%), followed by the without-catalyst sample (57.0%) and 10Ce-EA (39.1%). This indicates that the presence of the 10Fe-EA catalyst enhanced the cracking of CFW and PPW into lighter compounds. The increase in hydrocarbon composition is due to the catalyst's ability to deoxygenate the pyrolysis vapor, thereby producing low-oxygenated bio-oil [29]. Chen et al. [30] investigated the catalytic co-pyrolysis of bamboo and polyethylene under microwave irradiation. In this study, the iron-carbonized MOF (Fe@C) catalyst was observed to reduce oxygenates, especially acids and ketones, suggesting that the Fe@C catalyst facilitates decarbonylation, decarboxylation, and similar reactions. Conversely, 10Ce-EA produced lower total hydrocarbons compared to the one without a catalyst due to blockage of active sites located in pores, as evidenced by its low surface area and pore volume. In addition, iron oxide (FeO_3) was found to suppress the formation of cellulose and hemicellulose derivatives (oxygenates compound) compared to cerium oxide (CeO_3), resulting in the increment of total hydrocarbon of 10Fe-EA [31].

The %DOD revealed the conversion of oxygenates via secondary reactions using the 10Fe-EA catalyst, at 25.8%, while 10Ce-EA produced more oxygenated compounds than without a catalyst (-34.2%). Table 2 shows the hydrocarbon compositions of the bio-oils from 10Ce-EA, 10Fe-EA, and the no-catalyst control. Most of the hydrocarbon produced contains cycloalkane and alkene.

Table 2: Hydrocarbon composition of bio-oil using 10Ce-EA, 10Fe-EA and without a catalyst.

Sample	Without catalyst	10Ce-EA	10Fe-EA
Alkane	5.1	5.7	0.4
Alkene	32.5	24.5	32.5
Cyclo-alkane	62.5	69.8	67.1
Aromatic HC	0.4	0.0	0.0

The predominance of cycloalkanes over alkenes was observed in both catalytic and non-catalytic co-pyrolysis of CFW and PPW, indicating that the thermal degradation behavior of the feedstocks primarily governed the hydrocarbon distribution. During co-pyrolysis, hydrogen-rich radicals generated from PPW stabilize biomass-derived radicals through hydrogen transfer, while olefin intermediates undergo thermal cyclization to form cycloalkanes [32]. The Fe-EA catalyst did not substantially alter the hydrocarbon distribution but enhanced the overall hydrocarbon yield by promoting secondary cracking and deoxygenation of oxygenated vapors. These findings suggest that the synergistic interactions between biomass and plastic determine the dominant hydrocarbon species, whereas the catalyst mainly improves the selectivity of hydrocarbon production. Similar synergistic effects between cellulose and polypropylene have been reported by Ojha & Vinu [33], who attributed the enhanced hydrocarbon production to hydrogen transfer from polypropylene to biomass-derived radicals during non-catalytic co-pyrolysis.

4. CONCLUSIONS

In conclusion, 10 wt.% Ce and Fe-incorporated extracted aluminum (EA) from industrial sludge (10Ce-EA and 10Fe-EA) were successfully synthesized via the wet impregnation method. The catalytic activities of 10Ce-EA and 10Fe-EA were evaluated for the catalytic co-pyrolysis of cotton fabric waste (CFW) and polypropylene plastic waste (PPW). The findings reveal that catalytic activity is strongly influenced by the type of active metals and the resulting physicochemical properties of the catalyst. The 10Fe-EA catalyst exhibits superior catalytic performance owing to its favorable textural characteristics, which enhance interactions between pyrolysis vapors and the catalyst and promote bio-oil upgrading. This study highlights the potential to recover aluminum from industrial sludge as a sustainable catalyst support, offering an alternative route for waste valorization and reducing dependence on conventional catalyst materials. The enhanced hydrocarbon selectivity achieved with the Fe-based catalyst further demonstrates its

potential to produce higher-quality bio-oil from mixed cotton fabric and polypropylene waste. Future work should investigate the optimization of metal loading and reaction conditions, and evaluate the catalyst performance in continuous pyrolysis systems.

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