Synthesis of Bio-Oil via Catalytic Co-Pyrolysis of Cotton Fabric Waste and Polypropylene Plastic Waste

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ARTICLE INFO	ABSTRACT
	The growing energy demand and rising environmental concerns pose severe difficulties for future generations in maintaining energy sustainability. Therefore, it is critical to explore alternative green energy sources. Hence, this study synthesized renewable bio-oil by utilizing biomass of cotton fabric waste (CFW) and polypropylene plastic waste (PPW) as feedstocks. Titanium and alumina extracted from industrial solid waste (Ti-EA) were selected as catalysts due to their active sites promoting co-pyrolysis reaction towards bio-oil formation. The Ti-EA catalyst was prepared via the wet impregnation method at different calcination times. The bio-oil was synthesized via co-pyrolysis of CFW and PPW in a fixed bed reactor at the reaction temperature of 600 °C for 1 h. The effects of the catalyst calcination time were evaluated based on the bio-oil yield and product distribution. Moreover, the catalysts were characterized by BET and FTIR. The findings show that the Ti-EA catalyst calcined at 4 h (Ti-EA-4) obtained the highest bio-oil yield of 68% whereas calcination at 6 h (Ti-EA-6) gave the lowest yield of 28%. The BET shows that the Ti-EA-4 catalyst has a higher surface area of 121.6 m2/g than the Ti-EA-6 catalyst of 96.74 m^2/g. This indicates that surface area is an important characteristic that promotes efficient catalytic activity for bio-oil production. Meanwhile, FTIR results show that the catalysts have similar surface functional groups mainly O-H stretching vibrations. Overall, Ti-EA catalyst calcined at 4 h has exhibited good performance in producing bio-oil via co-pyrolysis of CFW and PPW. Thus, Ti-EA catalyst has the potential to produce bio-oil as fuel, which contributes towards waste to energy conversion and environmental preservation.

1. Introduction

The growing energy demand and rising environmental concerns pose severe difficulties for future generations in maintaining energy sustainability. Coal is the main source of the world's energy consumption, derived from fossil fuels. However, according to the United States Energy Information Agency, coal is predicted to be available until at least 2112, while the world's energy demand will rise

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by 56% in 2040 [1]. Therefore, suitable alternative energy is needed to overcome this major concern. Bio-oil, derived from renewable resources like biomass, can provide similar energy to fossil fuels and positively influence environmental attributes. The biomass feedstock can be generated from a variety of sources, including plants, animals, and wastes. Over the years, global fibre consumption has gradually increased due to the growth of population, increased standard of living, and the influence of fast fashion. Consequently, the quantity of textile waste has increased with the majority of the waste being disposed of [2]. Cotton fabric is among the most produced fibre-based textiles globally, with an annual production of over 50 million tonnes. The textile products that become waste eventually end up in landfills or incinerated, causing environmental pollution. Cotton fabric waste (CFW) mainly consists of cellulose with a small amount of pectin and protein, constituting a suitable bio-oil feedstock [3]. Polypropylene is the largest plastic kind that can be found in municipal solid waste (MSW) worldwide [4]. Polypropylene plastic waste (PPW) can be co-feedstock in bio-oil production due to the significance of hydrogen sources. Thus, CFW and PPW can be utilized to produce bio-oil as a green fuel that is renewable and environmentally friendly.

Co-pyrolysis of biomass and plastic waste is a promising technique to convert the mixture of polyolefins derived from plastics and cellulose or lignin derived from biomass into bio-oil. This process offers many advantages, such as a reduction in waste volume and production costs, expanded waste disposal alternatives, recovery chemicals, and reduced environmental impacts [5,6]. This technique is an easy and safe process that produces high-quality oil and does not lead to high-pressure hydrogenation [6,7]. The co-pyrolysis process is operated in a closed reactor in the absence of oxygen at the ranges of 400- 700 °C and under atmospheric pressure [8]. Bio-oil has several issues due to its high-water composition between 15% and 30%. The oxygenated compound and water content in bio-oil have negatively impacted fuel characteristics such as reducing combustion rates, enhancing corrosiveness, prompting phase separation, and instability, and reducing the heating value [9,10]. Hence, using a catalyst is important to promote the co-pyrolysis reaction and upgrade the quality of bio-oil.

Catalysts play a major role in co-pyrolysis to reduce the coke formation and increase the hydrocarbon content [9]. Catalysts can reduce the oxygen content, significantly reducing energy input and tar formation [11]. In recent studies, metal oxides such as TiO_2 and/or Al_2O_3-based catalysts have been widely used in oil upgrading through catalytic pyrolysis and co-pyrolysis due to their good catalytic activity, high stability of chemical, and require a small amount of catalyst for catalysis [12,13]. Industrial solid waste is extensively utilized in various applications due to its high content of precious metals such as aluminium (Al), ferum (Fe), and silica (Si). Most of them can be utilized as catalysts in co-pyrolysis, and they are economical and contribute to lower production costs [14]. Recent studies have demonstrated that several industrial solid wastes such as coal fly ash [15], steel slug [16], red mud [17], aluminium dross [18], and electrical arc furnace slag [19] have been applied as catalysts in bio-oil production. However, no studies have been reported on the co-pyrolysis of biomass and plastic utilizing titanium and alumina extracted from industrial solid waste as a catalyst and the effects of catalyst calcination time. Therefore, an attempt has been made in the present paper to study the synthesis of bio-oil via Ti-EA catalyst-assisted co-pyrolysis.

Therefore, this paper makes a significant contribution through its examination of the synthesis of bio-oil via catalytic co-pyrolysis of cotton fabric waste (CFW) and polypropylene plastic waste (PPW). The Ti-EA was prepared via the wet impregnation method at different calcination times. The catalytic co-pyrolysis process was conducted in a fixed-bed reactor. The effects of the catalyst calcination time were evaluated based on the bio-oil yield and product distribution. Moreover, the catalysts were characterized by BET and FTIR.

2. Methodology

2.1 Materials and Chemicals

Cotton fabric waste (CFW) was collected from a textile manufacturer in Penang, Malaysia, and polypropylene plastic waste (PPW) was gathered from a nearby residential area. Aluminium sludge waste was collected from the aluminium substrate industry in Penang, Malaysia. The chemicals used for Ti-EA catalysts preparation were titanium (IV) butoxide (97% purity), which was purchased from Sigma-Aldrich Malaysia Sdn. Bhd. Hydrochloric acid (37% purity) and acetone (99% purity) were purchased from QreC (Asia) Sdn. Bhd. Ammonia Hydroxide (30% purity) was purchased from R&M Chemicals Sdn. Bhd. Purified nitrogen, helium, and purified air were purchased from Alpha Gas Solution Sdn. Bhd. All the chemicals used were analytical reagent grade.

2.2 Preparation of Ti-EA

The extracted alumina (EA) was recovered from aluminium sludge waste by using the acid-leaching method. First, sludge waste was dried for 12 h in the oven at 110°C to remove the moisture content. Then, the dried sludge was leached with 4M hydrochloric acid in a ratio of sludge to the acid of 1:5 for 1 h at 70°C with a stirring rate of 250 rpm. The beaker was covered with aluminium foil to prevent the spilling of chemicals during the reaction. Then, the solution was filtered and aged overnight until the cloudy solution was obtained. Precipitation was performed by the addition of 10% ammonia solution dropwise until it reached pH 9. The white precipitate was further stirred for 30 min, and subsequently, the precipitate was centrifuged to separate from its solution. Then, washed with deionized water to remove excess ammonia solution content and dried in the oven at 110°C overnight. Finally, the dried extracted alumina (EA) was then calcined at 600°C for 2 h.

Ti-EA catalyst was prepared with Ti-EA via wet impregnation method. The typical catalyst preparation process of Ti-EA was as follows: 3 g of EA was dissolved in a 250 mL beaker containing 10 mL of distilled water. Next, 3000 mL titanium (IV) butoxide is slowly added into the same beaker, and the solution is magnetically stirred at 500 rpm for 4 h on a hot plate at 40°C to achieve uniform mixing. The sample is then dried in an oven for 12 h at 110°C, followed by calcination in a muffle furnace at 600°C and time (ranging from 1 h to 6 h).

2.3 Catalytic Co-Pyrolysis CFW and PPW

Catalytic co-pyrolysis of cotton fabric waste and polypropylene plastic waste was done in a fixed bed reactor under an inert condition as shown in Figure 1, where the reactor consists of two segments separated with the quartz wool. The first segment contained the feedstocks (CFW and PPW) while the second segment contained the catalyst. A k-type thermocouple was placed in the reactor to determine the temperature. The nitrogen gas was used at a flow rate of 70 mL/min at a heating rate of 10°C min^{-1}. The catalytic co-pyrolysis of 0.5 g CFW and 0.5 g PPW with 1 g Ti-EA catalysts was carried out at 600°C for 1 h. The condensed liquid oil was collected at the wax trap, and the gas was diverted to online GC-FID/TCD to capture the gas composition immediately. The yield of product was determined as Eq. (1) to Eq. (3) [20].

$$\text{Yield of liquid (wt.\%)} = \frac{\text{Liquid mass (g)}}{\text{Total mass (g)}} \times 100\% \tag{1}$$

$$\text{Yield of solid (wt.\%)} = \frac{\text{Solid mass (g)}}{\text{Total mass (g)}} \times 100\% \tag{2}$$

$$\text{Yield of gas (wt.\%)} = \frac{\text{Total mass (g)-Liquid mass (g)-Solid mass (g)}}{\text{Total mass (g)}} \times 100\% \qquad (3)$$
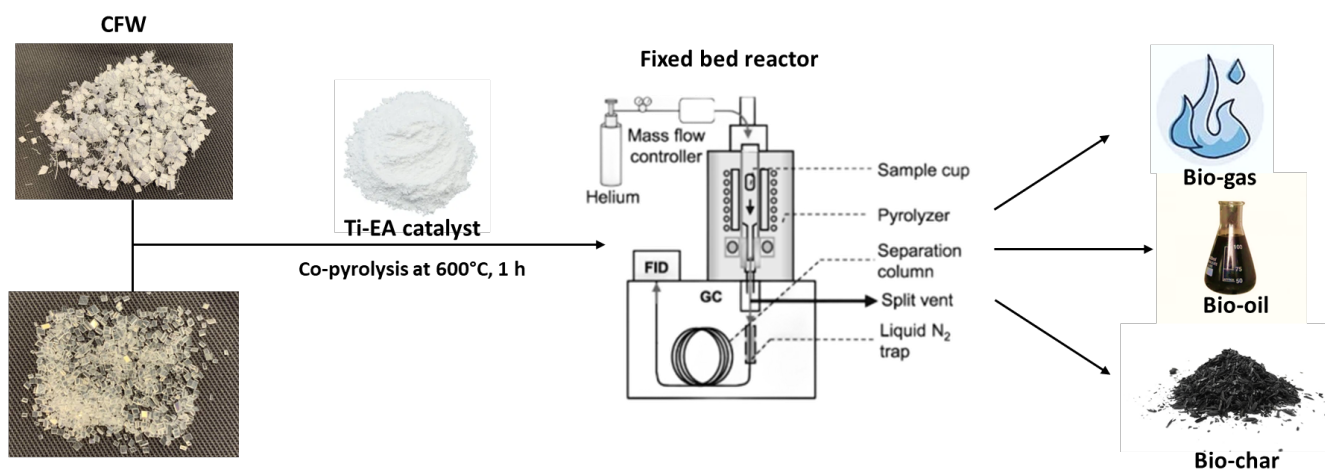

Fig. 1. Schematic diagram of the catalytic co-pyrolysis of CFW and PPW

2.4 Brunauer-Emmett-Teller (BET) Surface Area Analysis

The Brunauer-Emmet-Teller (BET) was used to evaluate the specific surface area, pore volume, and pore size distribution properties of the synthesized Ti-EA catalysts. This analysis was performed by using ASAP 2020 Micromeritics with a nitrogen adsorption/desorption analyser. N_2 adsorption and desorption on the catalyst surface were carried out at -196 °C in a vacuum chamber. The samples were degassed at 150 °C overnight to eliminate foreign gases and moisture from the catalyst surfaces.

2.5 Fourier Transform Infrared Spectrometry (FTIR) Analysis

Fourier transform infrared spectrometry (FTIR) was used to identify the functional groups of synthesized Ti-EA catalysts. This analysis was run by using Nicolet, Thermo Scientific spectroscope. Infrared radiation causes the vibrations and rotations of chemical bonds at specific frequencies that are special characteristics of a compound. The samples were characterized within the wavenumber range of 4000–400 cm^{-1}.

2.6 Gas Chromatography-Mass Spectrometry (GC-MS) Analysis

Gas chromatography-mass spectrometry (GC-MS) was used to determine the composition of bio-oil. This analysis was carried out using the Perkin Elmer Clarus 600/600 T, USA model with a capillary column of HP-5MS, operated at 40 °C for 2 min. Then, the temperature was ramped from 200 to 220 °C at a rate of 10°C min $^{-1}$ and maintained for 10 min. In this analysis, 0.2 g of bio-oil was diluted in 5 mL of acetone before it was transferred to the GC sample vial and injected into equipment via auto-injection mode for analysis.

3. Results
3.1 Brunauer-Emmett-Teller (BET) Surface Area Analysis

Table 1 shows the values of BET surface area, pore volume, and average pore diameter of Ti-EA catalysts. The findings show that Ti-EA-4 showed a higher surface area of 121.6 m^2/g, pore volume

of 0.53 cm^3/g, and average pore diameter of 175.07 Å compared to other Ti-EA catalysts. This indicates that 4 h calcination time is the optimum duration for calcination to produce good catalyst surface characteristics that promote better catalytic activity during co-pyrolysis reaction. More prolonged heat exposure during calcination leads to lower BET surface area due to the sintering effect that causes the porous catalyst structure to collapse. As reported by Wan *et al.,* [21], sufficient calcination time leads to the formation of uniformly distributed metal oxides with many active surface sites. Ti-EA catalysts are categorized as mesoporous since their average pore diameter sizes are between 20 and 500 Å [22]. The large pore allows large molecules, such as lignin-derived compounds, to flow in and out of the catalyst's pore, resulting in higher conversion of the compounds during the catalytic co-pyrolysis [23]. Therefore, Ti-EA calcined at 4 h gave a good catalytic activity performance in producing bio-oil with 68% yield.

Table 1
BET surface area, pore volume, and average pore diameter of Ti-EA catalysts

Catalyst	BET surface area (m^2/g)	Pore Volume (cm^3/g)	Average pore diameter (Å)
Ti-EA-1	109.33	0.48	171.55
Ti-EA-2	115.93	0.41	139.72
Ti-EA-3	97.43	0.44	178.11
Ti-EA-4	121.6	0.53	175.07
Ti-EA-5	83.48	0.39	184.40
Ti-EA-6	96.74	0.44	183.58

3.2 Fourier Transform Infrared Spectrometry (FTIR) Analysis

Figure 2 shows the FTIR spectra of Ti-EA catalysts at different calcination times in the region of 400-4000 cm^{-1}. The findings demonstrated that the spectra exhibited similar patterns that indicate common functional groups. The region 3800-3650 cm^{-1} represents O-H stretching vibrations involving hydrogen bonding in water molecules. Other peaks are found at 1650-1580 cm-1, which is the vibration of surface hydroxyl groups attached to the surface metals. Other peaks are found at 1190-900 cm^{-1}, which correspond to M-O-H stretching vibrations, while those in the 650-400 cm^{-1} range are attributed to M-O bending vibrations [21] (where M denotes the metal component of Ti and extracted alumina). Based on the observation, the peak is less pronounced in the Ti-EA catalyst calcined at 1 h compared to 6 h. This is because longer calcination time can lead to new compounds or phases forming, which can be reflected in the FTIR spectrum as new absorption bands [24].
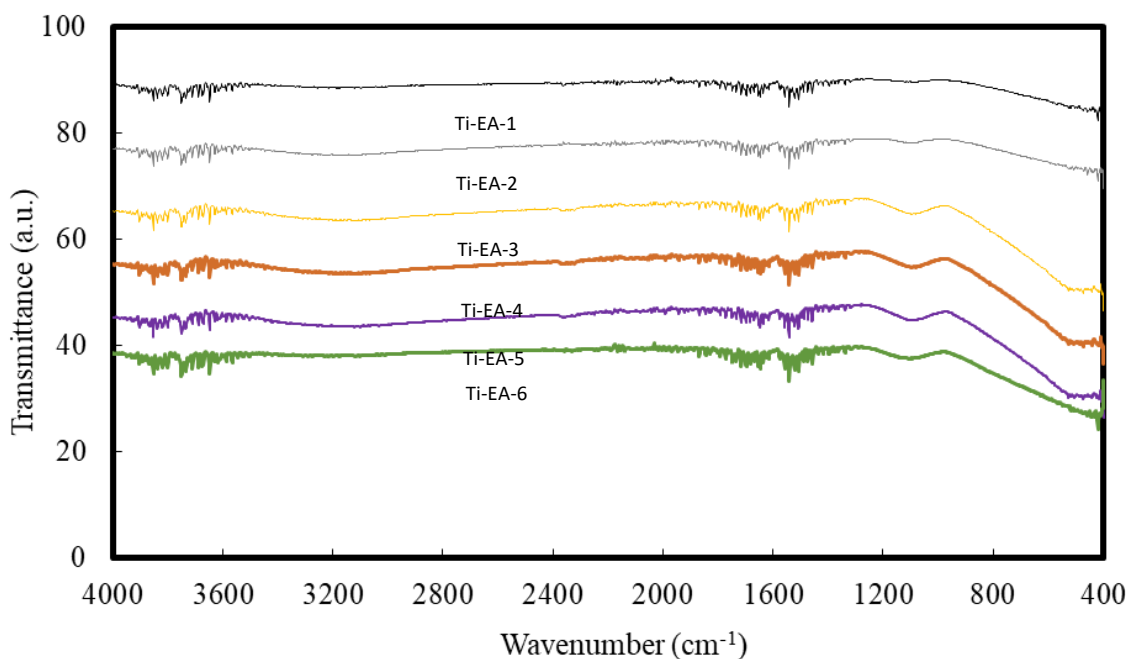
Fig. 2. FTIR of Ti-EA catalysts at different calcination time

3.3 Product Yield

Figure 3 depicts the product yield of Ti-EA catalysts at different calcination time. The result shows that the Ti-EA catalyst calcined at 4 h obtained the highest bio-oil yield, up to 68%, and the least of char and gas yield, which are 11.7 % and 16.9 %, respectively. On the other hand, Ti-EA calcined at 6 h gave the highest gas yield and lower char yield, which are 52 % and 18 %, respectively. However, the bio-oil yield at Ti-EA-6 shows the lowest yield, only 28%.   It is deduced that the Ti-EA-6 is active in the cracking process. However, it has a lower surface area, which improves the formation of gas hydrocarbons rather than liquid hydrocarbons during the catalytic cracking of CFW and PPW [25]. Thus, Ti-EA-4, with the largest surface area, showed a higher conversion of bio-oil yield than Ti-EA-6. This indicates that surface area is a significant factor in promoting efficient catalytic activity for bio-oil production.
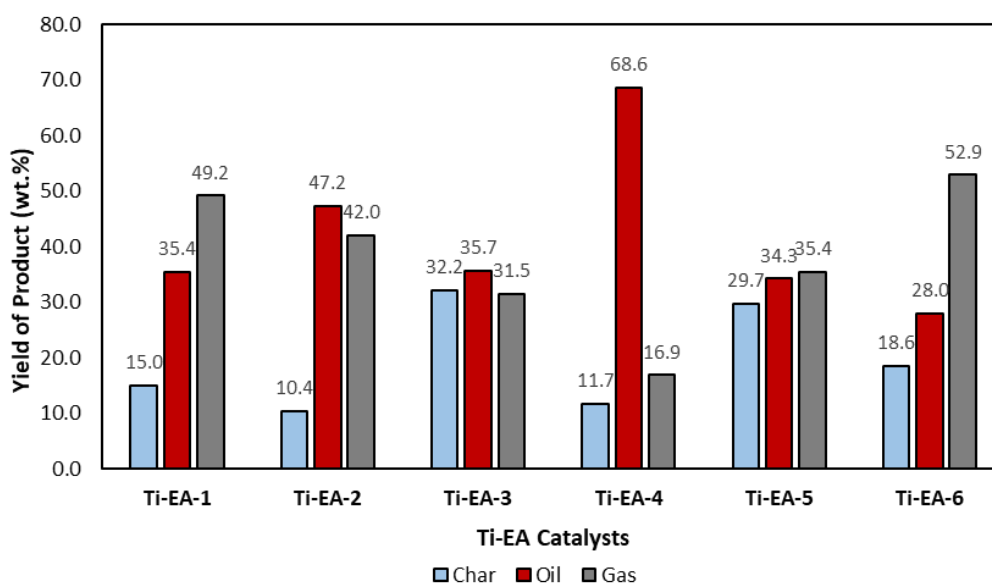

Fig. 3. Yield of product obtained at different calcination times

3.4 Gas Chromatography-Mass Spectrometry (GC-MS) Analysis

Figure 4 shows the product distribution of bio-oil via gas chromatography-mass spectrometry (GC-MS). The hydrocarbons in the bio-oil are significantly increased, as shown by an increased calcination time, Ti-EA-1 to Ti-EA-4, up to 51%-54%. However, as calcination time increased to Ti-EA-5 and Ti-EA-6, the hydrocarbon content was reduced due to the sintering effect of the Ti-EA catalyst at a longer calcination time. The oxygenated compounds such as acid, alcohol, ketone, and ester undergo deoxygenation reactions via dehydration, decarbonylation, and decarboxylation to form hydrocarbons [26]. The active sites of Ti-EA catalysts favour dehydration reaction over decarbonylation and decarboxylation reaction, resulting in the removal of oxygen from the bio-oil and its subsequent combination with hydrogen to generate water as a by-product [27]. Therefore, Ti-EA calcined at 4 h shows a stable and good catalytic activity that leads to the formation of a higher content of hydrocarbons (54%) compared to Ti-EA calcined at 6 h (39%) from the catalytic co-pyrolysis of CFW and PPW.
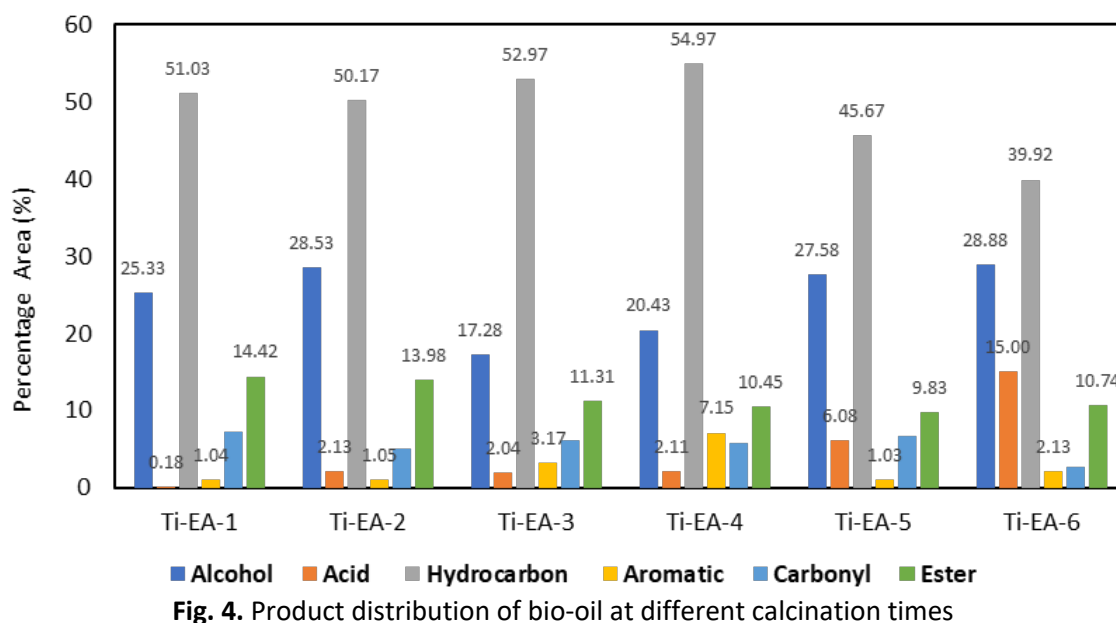

Fig. 4. Product distribution of bio-oil at different calcination times

4. Conclusions

Catalytic co-pyrolysis is one of the effective ways to convert waste from biomass and plastics into value-added product bio-oil. The study on the effects of the calcination time of Ti-EA catalysts on co-pyrolysis of CFW and PPW shows that Ti-EA-4 obtained bio-oil with the highest yield of 68%. Moreover, the highest hydrocarbon content was Ti-EA-4, with a percentage of 54.97%. The results show higher hydrocarbon and lower bio-oil oxygen content than Ti-EA-6, indicating successful dehydration, decarboxylation, and decarbonylation reactions during co-pyrolysis. The BET shows that the Ti-EA-4 catalyst has a higher surface area of 121.6 m^2/g, pore volume of 0.53 cm^3/g, and average pore diameter of 175.07 Å. Overall, Ti-EA catalyst calcined at 4 h has exhibited good performance in producing bio-oil via co-pyrolysis of CFW and PPW. Therefore, Ti-EA catalyst has a good potential for bio-oil production as fuel, contributing to waste-to-energy conversion and environmental preservation.

Acknowledgement

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