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Extraction of K_2CO_3 from empty palm fruit bunch ash and properties analysis

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Abstract

Empty oil palm fruit bunches (EPFB) are the largest solid waste generated from palm oil mills. Accounting for 21-23 % of the total fresh fruit bunches. EFB ash contains K (Potassium) which can be recovered as K_2CO_3 through extraction. This research aims to recover K_2CO_3 from empty oil palm fruit bunches (EPFB) as a catalyst raw material because it is alkaline using the K_2CO_3 extraction method from the ash of empty oil palm fruit bunches (EPFB), which begins with a combustion process at a temperature of 700 ^oC to obtain ash. Ash extraction was carried out in 2 stages using water as a solvent. The K_2CO_3 yield from empty palm fruit bunch ash reached 57.28 %. The alkalinity test showed that the K_2CO_3 content in the solid extract reached 85.91 %. The properties of the ash and K_2CO_3 produced were analyzed using XRF, XRD and FTIR. The results of XRF analysis show that the potassium content in the ash contains the element K (potassium) which is quite large, namely 71.24 % and after extraction the purity of potassium oxide reaches 97.08 %. XRD analysis of the results of the synthesis of potassium carbonate from empty oil palm fruit bunches showed that the intensity of the material beam was obtained in a 2ϴ pattern which was lower than the intensity pattern of pure potassium carbonate. The results of FTIR analysis show that the spectrum of carbonate ions from the EPFB C-O bend is visible at 1359,879 and 700.75 cm-1 and these results are similaire to the pure carbonate ion vibration mode.

Keywords: ash, empty palm fruit bunches, extraction, potassium carbonat

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1. INTRODUCTION

Indonesia is the largest producer of palm oil in the world, with total CPO production in 2023 of 50.07 million tonnes/year with an abundance of solid waste from empty palm fruit bunches of 15 million TON EPFB/year (Arita S. et al., 2023). According to Nyakuma et al., (2020) the palm oil industry produces quite a lot of solid waste in the form of EPFB compared to other solid waste such as palm shells and

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palm fiber. In palm oil plantations, most of the solid waste processed as conventional fertilizer is spread directly on the plantation land, which over time will rot and automatically fertilize the plantation land. Apart from that, the solid waste becomes material for filling roads and as pavers.

Most researchers utilize empty oil palm fruit bunches (EPFB) waste into compost (Thambirajah J.J. et al., 1995; Dayana et al., 2011; Baharuddin et al., 2011; Kabbasi et al., 2014; Shafiquzzaman et al. , 2017 and Schucharde F. et al., (2005) which combined EFB and POME in making compost fertilizer. The use of EFB in the paper mill sludge processing mixture was carried out by Rosazlin et al., (2011) and Kananam et al., (2011) looking at the biochemical changes in compost fertilizer resulting from a mixture of EPFB with sludge and chicken manure. Life cycle inventory of the commercial production of compost from oil palm biomass has been studied by Norhasmillah et al., (2013) and Zahrim et al., (2010).

Empty oil palm fruit bunches (EPFB) are also widely used as raw material for making active carbon to manage industrial liquid waste and absorb various dyes (Tan et al., 2009; Firoozian et al., 2011; Sajab et al., 2013; Alya et al., 2011; al., 2016; Osman et al., 2016; Norhidayah et al., 2016 and Ismail et al., 2018). The production of chemical compounds such as lignophenol, citric acid and sucrose fermented from empty oil palm fruit bunches (EPFB) was studied by Abdullah et al., (2009); Alam et al., (2010) and Ibrahim et al., (2015). The conversion of EFB into bio-oil through the pyrolysis and liquefaction processes of EFB was studied, among others, by Suchithra et al (2018); Yen Yee et al., (2020) and Sunaryo et al., 2023.

Potassium carbonate (K_2CO_3) is an inorganic compound that has been used as a base catalyst in various reactions. Apart from that, K_2CO_3 also has several benefits, namely as a source of potassium ions and buffer alkalinity due to its greater solubility (Kidwai et al., 2013). Potassium ions are more reactive than sodium ions, and can reduce the freezing point of water. Imaduddin et al. (2008) and Udoetok (2012) stated that oil palm empty fruit bunches (EPFB) contain quite a lot of K_2CO_3 so they can be developed as a source of catalyst raw material. This is supported by research by Sanjaya et al. (2017) who analyzed K_2CO_3 levels in EPFB ranging from 17-24%. Samadi et al., 2020 have succeeded in extracting potassium from biochar resulting from burning EPFB at a temperature of 400 ^oC using water as a solvent. The percentage of potassium produced reaches 45.5 %.

Several studies have shown success in potassium extraction. The percentage of extraction results is influenced by the solubility level of the material in the solvent, temperature, solvent contact time, and stirring. Water was chosen as a solvent because its ability to extract potassium is very large, where the highest solubility is obtained at a temperature of 90 ^oC. It is generally known that potassium compounds have very high solubility in water, due to the high hydration energy of K+ ions and the solubility of K_2CO_3 is almost 100 % (Samadhi et al. 2018; Oleszek et al., 2022; Behera et al., 2015; Boakye et al. al., 2018; Wang et al., 2014; Sanjaya et al., 2017; Sukeksi et al., 2017; Sitorus et al., 2018 dan Melani et al., 2021).

Potassium Carbonate has advantages, especially regarding the environment, non-toxic, physico-chemical properties and relatively low cost (Gohla et al., 2020). In its application, K_2CO_3 is widely used in various processes, including phenol alkylation, ester and amine hydrolysis, C/O alkylation, dehydrating agent, corrosion inhibitor, and catalyst for various reactions (Mondal et al., 2014). In this research, an attempt was made to extract K_2CO_3 from EPFB ash which will be used as a catalyst raw material for producing biodiesel because of its alkaline nature.

2. MATERIALS AND METHOD

Palm oil empty bunches (EPFB) come from the PTPN 7 South Sumatra palm oil mill. The initial stage of extraction of empty oil palm bunches is pre-treatment of EPFB which includes drying and reducing the size of the EPFB then burning the EFB, the ash from the combustion is then sifted to produce the same size of ash, namely 100 mm, after that the EPFB ash is calcined at a temperature of 700 °C for 5 hours. The calcination temperature is determined based on the melting point of potassium which is 745 °C. It is hoped that at a temperature of 700 °C potassium will not melt and evaporate during calcination. The profile of EPFB ash before and after calcination can be seen in Figure 3 and the stage of the process from raw material retreat to the extraction process can be seen in the figure 1.

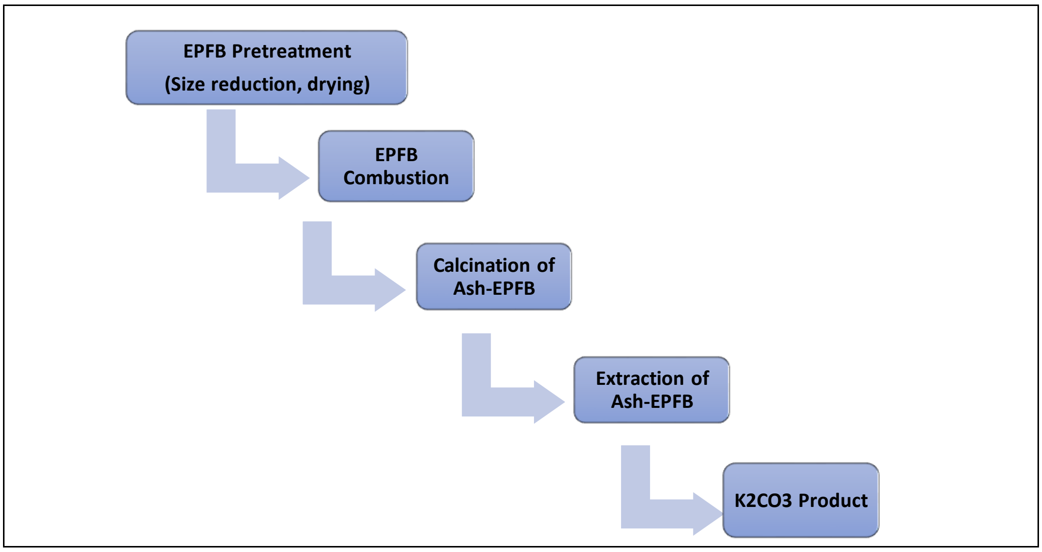

Figure 1. Flow chart the stage of the process from raw material retreat to the extraction process

2.1. Potassium Extraction from empty oil palm bunch ash (EPFB)

The extraction method includes the process of contacting EFB ash with a solvent, filtration process, evaporation of the filtrate, and drying of the solids formed. The extraction scheme can be seen in Figure 2.

Figure 2. K_2CO_3 Extraction from empty oil palm bunch ash (EPFB)

50 grams of empty oil palm bunch ash (EPFB) was put into a glass beaker, 250 mL of distilled water was added slowly while stirring, then heated with a hot plate to 90 °C for 90 minutes, stirring was carried out at a speed of 350 rpm. Then the filtrate is filtered, then evaporated at a temperature of 100 °C for 5 hours to evaporate the water and obtain K_2CO_3 salt crystals, while the residue is taken for later stage 2 extraction. The K_2CO_3 salt obtained is then re-ovened at a temperature of 120 °C for 2 hours. until dry, finally, before storing it in the bottle, grind it with a porcelain crucible.

To determine the potassium and K_2O content in calcined ash, a potassium content test was carried out using XRF (X-Ray Fluorescence). X-Ray Fluorescence (XRF) is a non-destructive analytical technique used to identify and determine the concentration of elements present in solid, powder or liquid samples. In general, XRF measures the wavelength of individual material components from the fluorescent emissions produced by a sample when irradiated with X-rays (Fatimah, 2018).

The K_2CO_3 levels contained in the ash were analyzed using acid-base titration. XRD analysis is carried out to determine the diffraction pattern of x-ray interaction with scattered crystalline compounds according to the composition or type of crystal. The scattering (diffraction) pattern of X-rays is a characteristic of each compound that is independent of each other. The wavelength of X-rays used for (- Xray Difraction (XRD) ranges from 0.5 - 2.

3. RESULTS AND DISCUSSION

3.1. Combustion of empty oil palm bunches (EPFB)

Combustion of empty oil palm bunches (EPFB) at a temperature of 450-550 °C produces black ash which shows the amount of carbon contained in the ash as seen in Figure 3. The analysis results show that the potassium oxide (K_2O) compound in the charcoal is 38.08 %. In Alva's (2021) research, the potassium content in empty oil palm fruit bunches (EPFB) ranged from 11.83 – 25.14 %.

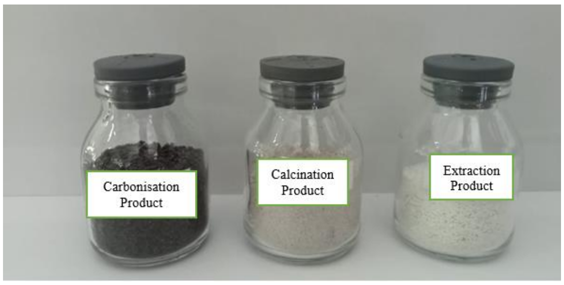

Figure 3. Product the Carbonisation, Calcination and Extraction Result

3.2. Calcination of Ash Palm Oil Empty Bunches (EPFB)

Calcination at a temperature of 700 °C produces whitish gray ash, indicating that the carbon element is starting to decrease. From the results of the XRF analysis, it shows that the dominant oxides in the ash are $K_2O > CaO > SiO_2 > Fe_2O_3 > P_2O_5 > ZnO > TiO_2$, the rest are other heavy metals with very small levels. This metal oxide is an alkali metal oxide which can support the effectiveness of EFB ash as a raw material for making biodiesel catalysts. The composition of metal oxide compounds and the results of the XRF graph can be seen in Figure 4.

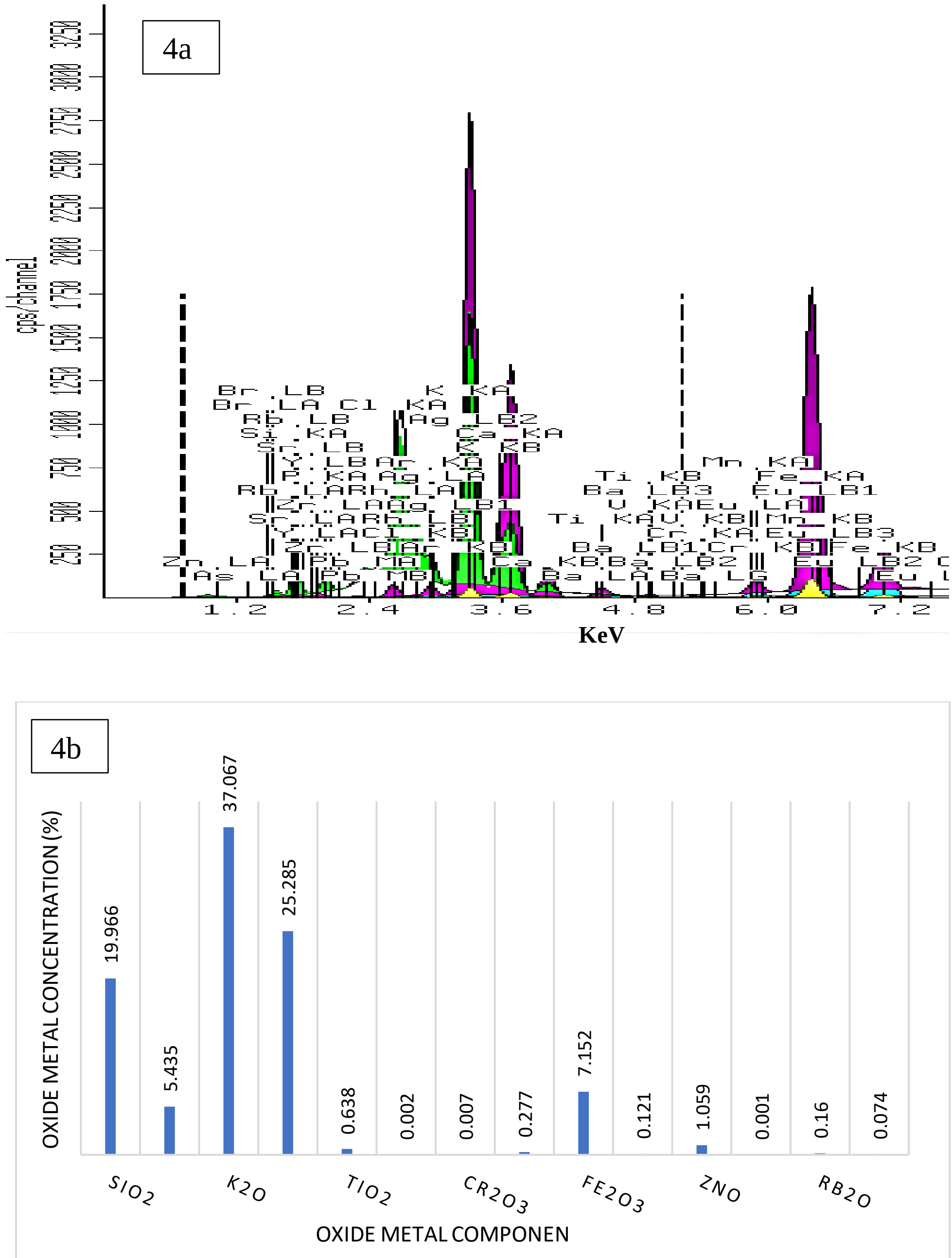

Figure 4. (a) graphs of XRF analysis results and (b) Composition of metal oxide components in ash Calcination results

Metal oxides in calcined EFB ash products are in the intensity range from 0 to 1250 cps/channel with the sum peak seen in the wave energy estimated at 9.7 KeV from 2 characteristic X-rays, namely 3.2 KeV and 6.5 KeV, as well as the potassium spectrum peak is found at an intensity of around 2900 cps/channel.

3.3. K_2CO_3 Extraction from empty oil palm bunch ash (EPFB)

The principle of extraction is dissolving the solute contained in the solid using a solvent (Aji et al., 2017). The solvent used must have the ability to dissolve the solute, in this case K_2CO_3. Several studies used various types of solvents to extract K_2CO_3. The methanol solvent was used by Imaduddin et al., (2008). In this research, water was used as a solvent. The water solvent was chosen because of the relatively high solubility of potassium in water. Potassium salts, especially K_2CO_3, are significantly more soluble in water and have higher basic strength in aqueous media than other alkali salts, such as sodium alkali (Malins, 2018). Apart from that, the solvent nature of water which is easy to obtain, in large quantities also aims to reduce the use of chemicals thereby creating a cleaner process.

The yield of potassium oxide produced from the extraction process is very high, namely 97.08 %, the graph in Figure 5 shows that the highest potassium yield is at the peak of the spectrum with an intensity of 8800 cps/channel and the sum peak can be obtained at wave energy with a magnitude of 6.8 KeV from 3, 2 KeV and 3.6 KeV. High light intensity indicates a high concentration of potassium oxide contained in K_2CO_3 salt crystals.

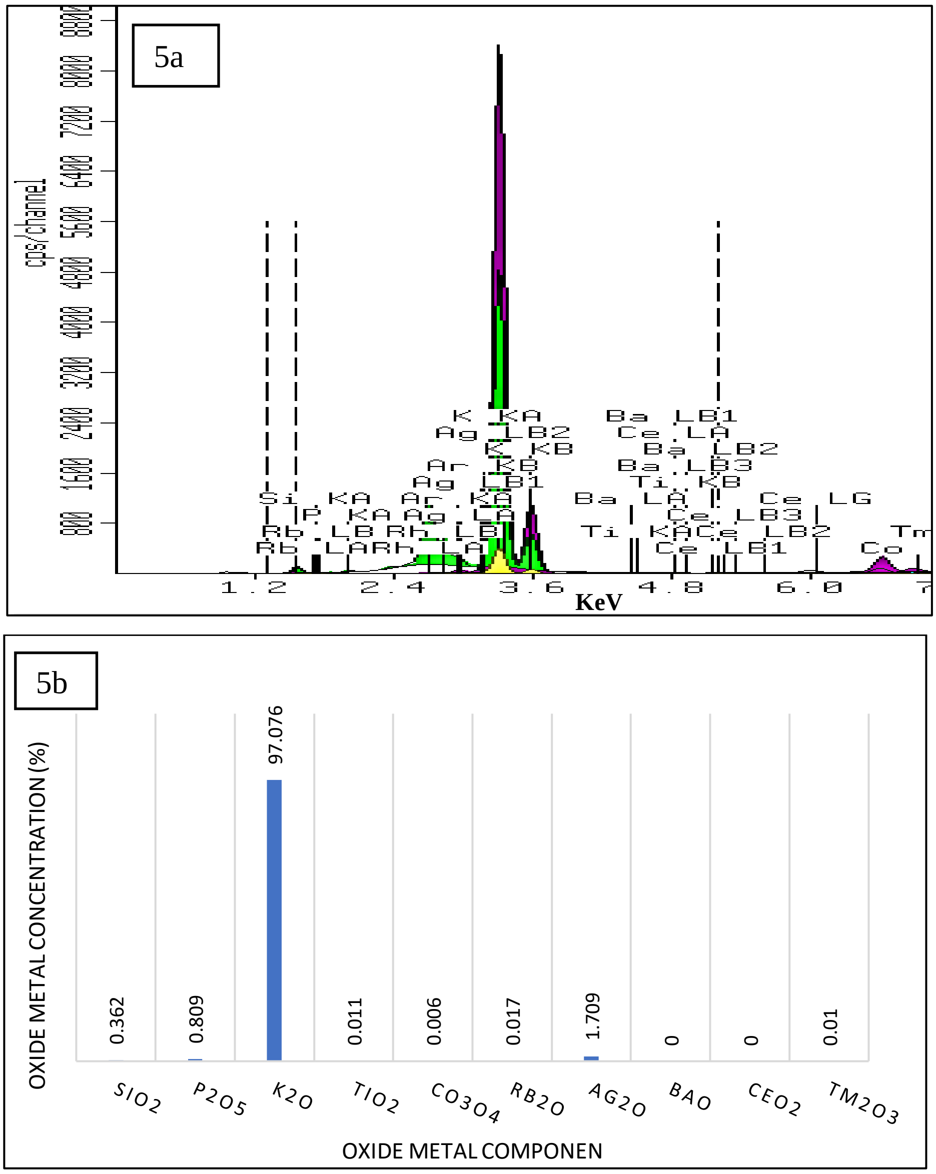

Figure 5. (a) graphs of XRF analysis results, and (b) Composition of metal oxide components in ash Extraction results

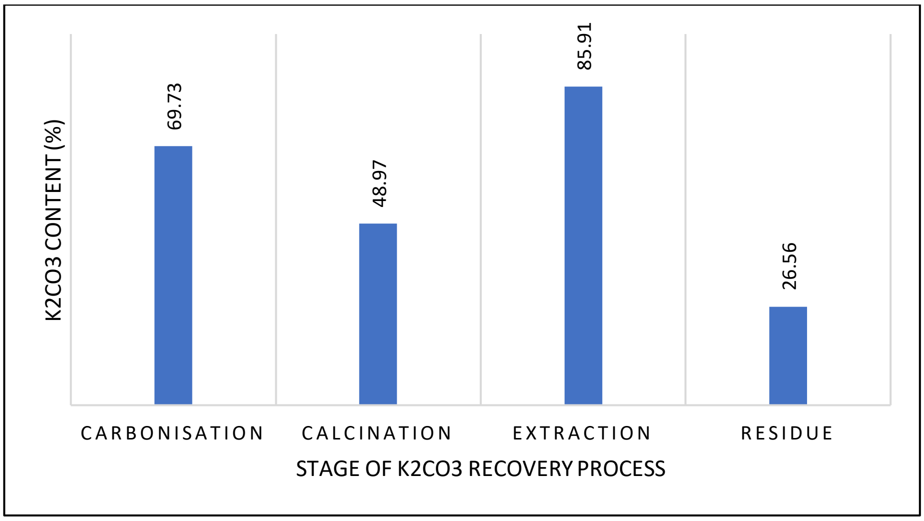

Figure 6. Purity of K_2CO_3 Results the Stage of Recovery Process (acid-base titration)

The percentage of K_2CO_3 was obtained from the results of analysis using the acid-base titration alkalinity method. With this alkalinity analysis, it is known that the purity of the extracted K_2CO_3 reaches 85.91 %, while the K_2CO_3 content resulting from carbonization is only 69.73 %. There was an increase in the percentage of K_2CO_3 levels after extraction. This is because the solubility level of potassium in water is very high, which is 156 g / 100 g of water. while Yitnowati et al. (2008) produced K_2CO_3 levels reaching 89.58 %. Shows that water is able to extract K_2CO_3 contained in empty oil palm fruit bunches.

The profile of the increase in K_2O content of empty palm fruit bunch ash (EPFB) from XRF analysis as shown in table 2 shows the K_2O content at each process stage. It can be seen that the raw material of Empty Palm Oil Bunches (EPFB) contains 48.21 % potassium oxide. When carbonized the percentage of all oxides increases where the K_2O content increases to 60.94 %. A carbonization temperature of between 450-550 °C is able to open the pores of Empty Palm Oil Bunches (EPFB) ash to a greater extent so that oxide compounds such as silica, sulfur and potassium oxide decompose more because their melting temperature has not been exceeded.

When continued with the calcination process at a higher temperature, namely 700 ^{o}C, all oxides increased but K_2O decreased to 35.65 %. The melting point of Potassium is 724 °C. With its hygroscopic nature, potassium evaporates at a temperature of 700°C, and this is explained by Okoye et al., 2019, that the decreasing nature of potassium levels occurs because calcination at too high a temperature causes potassium to also evaporate and this causes the K_2O content to decrease and the content to increase. other metal oxides during the calcination process.

Then a further process was carried out, namely extraction with a water solvent, all oxides were reduced and the reduction efficiency reached 99 % so that K_2O rose drastically to 97.08 %. For more details, see tabel1. When compared with the K_2O content in commercial K_2CO_3, which is 99.5 %, the extraction result from EPFB has quite a future to be used as a catalyst raw material in the transesterification reaction for biodiesel production because of its alkaline nature.

Tabel 1. The Compound of oxides in the K_2CO_3 process recovery

Compound of Oxides	Raw MateriaL EPFB (%)	Carbonisation (%)	Calcination (%)	Extraction (%)
SiO_2	15,90	20,25	35,02	0,36
P_2O_5	5,24	5,207	6,53	0,81
SO_3	1,98	3,25	4,65	0
K_2O	48,21	60,94	35,65	97,08
CaO	13,65	0	8,71	0
Fe_2O_3	7,22	0,85	1,97	0
TiO_2	0,79	0,31	0,02	0,01
Ag_2O	0	0	0	1,71

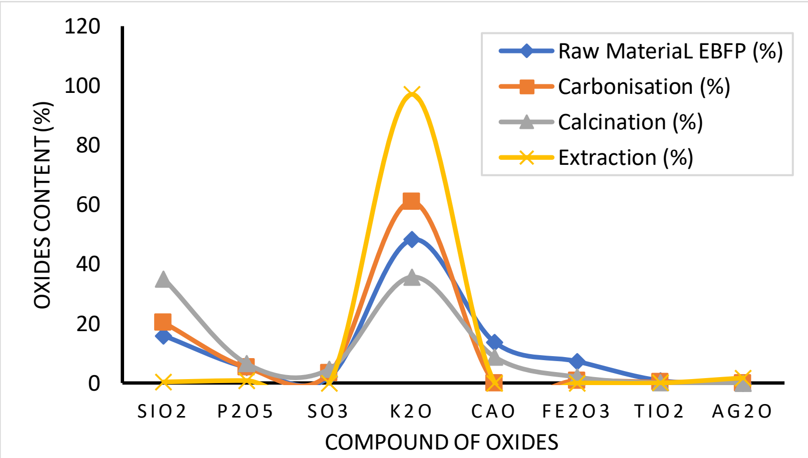

Figure 7. K_2O levels at the carbonization, calcination and extraction process stages (Analysis with XRF)

3.4. XRD Analysis

XRD analysis is intended to see the comparison of interference that produces the beam intensity pattern scattered from pure K_2CO_3 powder material and extracted K_2CO_3 on a scale of ±1Å. From the graph, it can be seen that the intensity of the two materials was obtained in different 2ϴ patterns as shown in table 3. For K_2CO_3, the highest intensity pattern extraction results were obtained at 2ϴ- 28.76 o lower than the pure K_2CO_3 pattern, because the purity of the extracted K_2CO_3 is indeed lower.

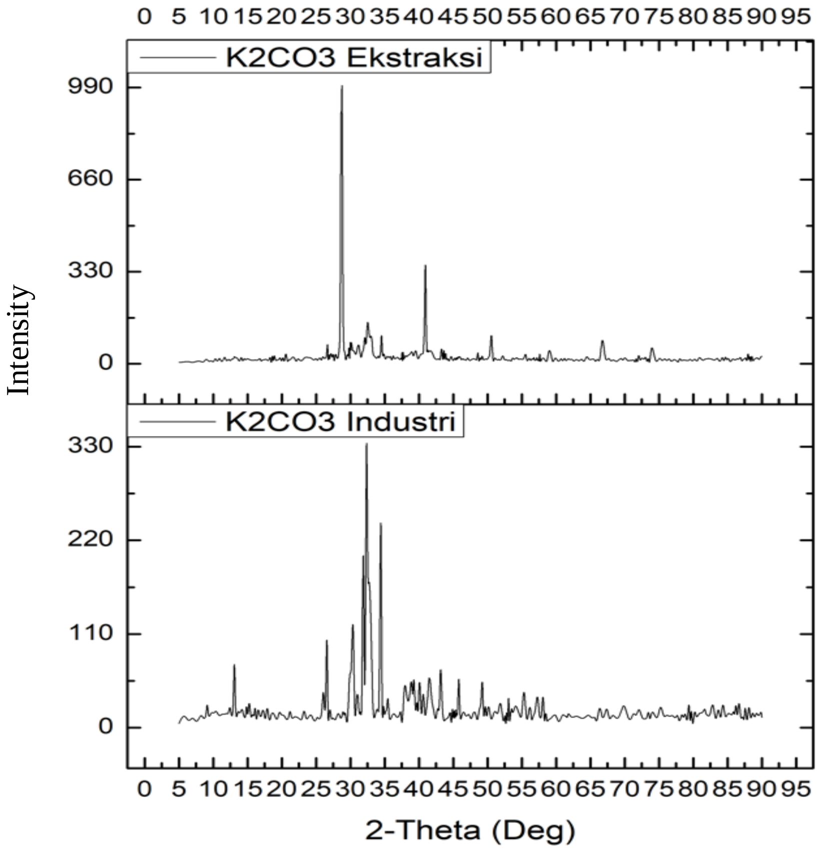

Figure 8. Comparison of XRD Analysis Results for K_2CO_3 extraction results and pure K_2CO_3

Compound	2Ө						
K2CO3 P	26,5^o	30,36^o	31,88^o	**32,36^o**	32,44^o	40,08^o	43,16^o
K2CO3 EPFB	26,6^o	**28,76^o**	32,52^o	34,54^o		40,96^o	50,58^o

3.5. FTIR Analysis

According to Yunlu Ma et al., (2021), carbonate ion spectra have 4 vibration modes. The first C-O vibration mode is symmetric at 1060 cm-1, usually the strain is strong and is least affected by most other species. Both out-of-plane deformation modes are 884 cm-1. The three C-O strain modes are anti-symmetric at 1395 cm-1 and the four deformation modes are in the 684 cm-1 plane.

The results of the FTIR analysis in Figure 9 are a comparison of the spectrum of carbonate ions originating from pure K_2CO_3 and K_2CO_3 extracted from empty oil palm fruit bunches. The carbonate ion spectrum from the EPFB C-O bend is seen at 1359, 879 and 700.75 cm-1. These results are close to the pure carbonate ion vibration mode where the C-O bend is seen at 1359.37, 879.34 and 689.2 cm^{-1}.

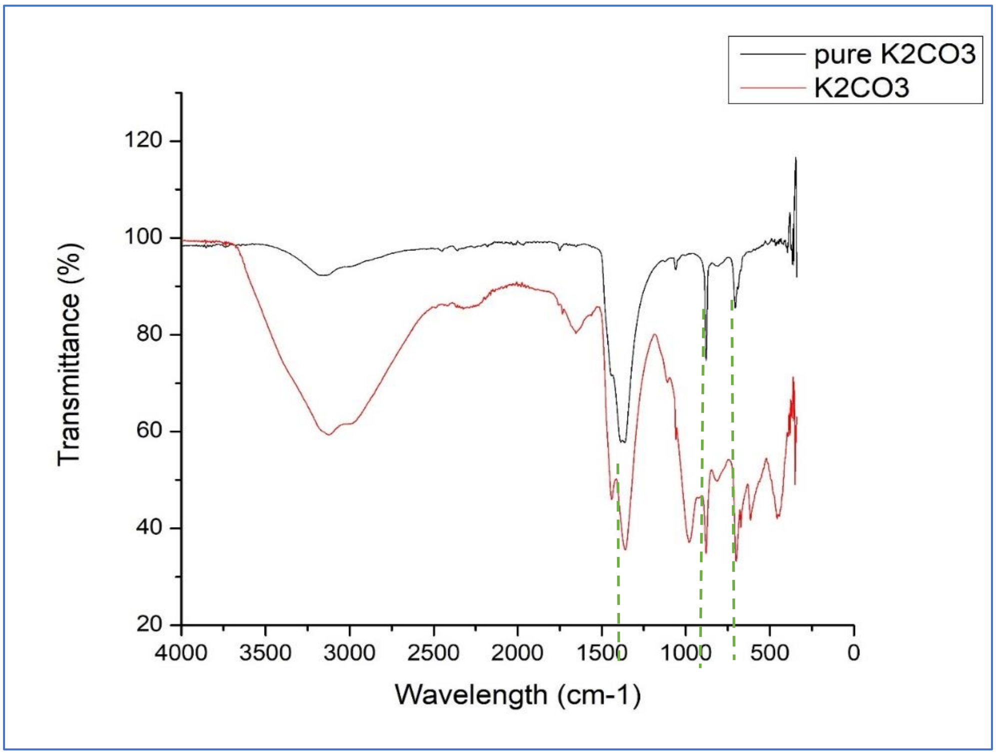

Figure 9: Comparison of XRF Analysis for K_2CO_3 extraction results and pure K_2CO_3

3.6. Mass Balance of K_2CO_3 Extraction from EPFB Ash

The mass balance calculation for the extraction of empty oil palm fruit bunches (EPFB) is intended to determine the yield of raw materials from EFB ash to K_2CO_3 and also determine the level of success of extraction in obtaining the desired components or compounds. The mass balance of EPFB ash extraction can be seen in Figure 10.

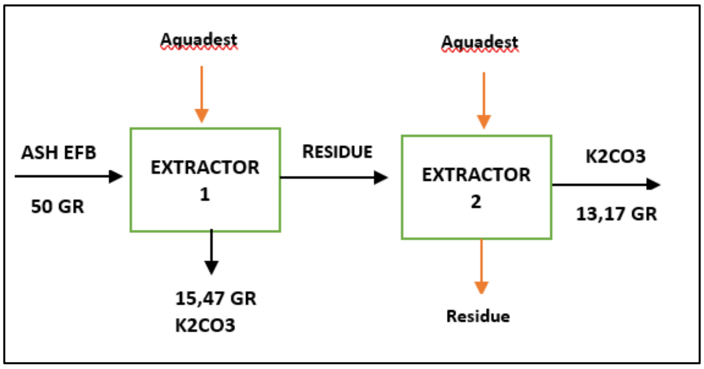

Figure 10. Mass balance of K_2CO_3 extraction result from EPFB ash

The mass balance of K_2CO_3 extraction from EFB ash can be seen in Figure 10. First stage K_2CO_3 extraction produced an average yield of 30.93 % of the total incoming EFB ash feed (per 50 gr). To increase the extraction yield, a 2-stage extraction was carried out. So, the total yield of K_2CO_3 from EPFB ash reached 57.28 %. However, in stage 2 extraction, the extraction results tend to be cloudier compared to stage 1, which is whiter in color. The results of EPFB ash extraction using water as a solvent were also carried out by Sanjaya (2017) with an average extraction yield of

24.50 %. This value is almost the same as the results of EPFB ash extraction using methanol solvent carried out by Imaduddin et al. (2008); Yitnowaty et al., (2008) where they obtained K_2CO_3 25.92 %. Meanwhile, Pambudi et al., (2019) tried to extract K_2CO_3 from boiler ash using a water solvent, they produced a K_2CO_3 yield of only 15.55 %.

4. CONCLUSION

The source of potassium can be obtained from empty oil palm fruit bunches through an extraction process with distilled water. The K_2CO_3 yield of 57.28 % was produced with 2 extraction stages. Analysis using the acid-base alkalinity system showed that the purity of the K_2CO_3 salt obtained was 85.91 %. The properties of ash and K_2CO_3 produced were analyzed using XRF, XRD and FTIR. The results of XRF analysis showed that the potassium oxide content in the ash after extraction reached 97.08 %. XRD analysis of the results of the synthesis of potassium carbonate from empty oil palm fruit bunches showed that the intensity of the material beam was obtained in a 2Ө pattern which was lower than the intensity pattern of pure potassium carbonate. The results of FTIR analysis show that the spectrum of carbonate ions from the EPFB C-O bend is visible at 1359, 879 and 700.75 cm-1 and these results are similaire to the pure carbonate ion vibration mode.

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