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Characteristic study of blended wastes for thermal process: Alum sludge mixed palm oil decanter cake for low slagging tendency

KUNMI Joshua Abioye[1,a], NOORFIDZA Yub Harun[1,b*], AFOLABI Haruna Kolawole[1,c] and AHMAD Hussaini Jagaba[2,d]

[1]Department of Chemical Engineering, Universiti Teknologi PETRONAS, 32610 Seri Iskandar, Perak, Malaysia

[2]Department of Civil and Environmental Engineering, Universiti Teknologi PETRONAS, 32610 Seri Iskandar, Perak, Malaysia

[a]kunmi_20001938@utp.edu.my, [b*]noorfidza.yub@utp.edu.my, [c]haruna_18002856@utp.edu.my, [d]ahmad_19001511@utp.edu.my

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Abstract. Proper and effective utilization of biomass wastes, couple with efficient conversion into useful energy have made the study of biomass wastes and finding solution to ash deposition during thermal conversion crucial. In this work, Alum Sludge (AS) was used to regulate ash deposition in Palm Oil Decanter cake (PODC). AS and PODC were characterized using Scanning Electron Microscopy and Energy Dispersive Spectroscopy (SEM-EDS), Thermogravimetric analysis (TGA), Advance X-ray diffractometer (XRD) while Ash fusion test (AFT) was used to determine the ash fusion temperature. SEM-EDS revealed the presence of Aluminum (13.50 wt%), Potassium (1.46 wt%), Iron (2.43 wt%) and Silicon (14.95 wt%) in AS while Potassium (1.19 wt%), Silicon (1.41 wt%), Calcium (2.16 wt%) and Chlorine (0.67 wt%) in PODC. TGA revealed that fixed carbon, volatile matter and ash content in AS are 8.94 wt%, 3.60 wt% and 86.72 wt% while PODC are 7.13 wt%, 51.84 wt% and 38.15 respectively. The total weight loss of AS and PODC was 13.29% and 61.86% respectively. XRD revealed the presence of Kaolinite and Quartz/ Zeolite in AS while Whewellite, Quartz, Potassium Chloride in PODC. AFT revealed 1215℃ and above 1400℃ as the sintering temperature for PODC and AS respectively. At 50% and above of AS addition, low slagging tendency of PODC was attained. This study provides a new insight on how AS and PODC wastes can be properly managed and utilized in clean energy production.

Introduction

Economic and population growth have led to an increase in the utilization of biomass. Biomass are utilized on daily basis and as a result, numerous biomass wastes are generated annually all over the world. Proper handling and management of this wastes have been a challenge as most end up on landfills and flowing streams or river. In Malaysia, over 2 million tons of Alum Sludge (AS) are generated per annum [1] as AS is an unavoidable waste from water purification and waste water treatment where alum salts are used during the coagulation process [2]. AS are generally known to be dominated with Aluminum and Silicon content, low organic and neutral pH [2]. When discharge into water bodies, it causes siltation, serve as potential risk to human health and disrupt the life of river biota due to the high presence of Aluminum and other metal contents. On the other hand, palm oil decanter cake (PODC); a palm oil biomass is a solid waste generated from the purification of crude palm oil, usually separated from liquid [3]. PODC comes from the mesocarp of oil palm fibers during processing at the mill. Malaysia is the second largest producer of palm oil with total production of 19,700 million tons which is equivalent to 26% of the total world palm oil production [4]. As a result, huge amount of PODC is generated in Malaysia. According to [5], each palm oil mill process 1,065 tons of fresh fruit bunch per day and one ton of fresh fruit bunch can produce PODC as much as 4.0% or as much as 40 kg. Mills dumped PODC around oil palm plantations or air burn thereby resulting in environmental pollution and hazards [6]. According to [7,8], PODC like other oil palm biomass have high potential for biofuel or bioenergy applications.

Both AS and PODC are biomass wastes which constitute nuisance to the environment when dispose improperly. According to [9], all kinds of biomass can be transformed into energy including starchy, triglyceride and lignocellulosic biomass. Biomass wastes can be converted to biofuels and biopower via thermochemical [7,10]and biochemical processes [11,12]. Thermochemical conversion produces products like bio-methanol, biodiesel, bio-oil, bio-syngas and biohydrogen while biochemical conversion produces liquid or gaseous fuels through fermentation or anaerobic respiration. The production of biofuels through thermochemical conversion processes with a broad range of technologies has drawn the most attention in the world [9] because of its instantaneous production and high-quality end products. However, the thermochemical conversion of oil palm biomass through gasification faces ash deposition issues [13] which affects the end product quality, reduces biomass gasification efficiency and cut short equipment lifespan. Oil palm biomass i.e., PODC, oil palm fronds, oil palm trunk are good source of energy as they possess high calorific value[12,14]. According to [13], ash deposition in biomass gasification is as a result of the presence of Alkali metals and Chlorine. Several researchers have worked on solving biomass ash deposition using Aluminosilicate based additives such as Kaolin/ Kaolinite and Zeolite. [15] reported that Kaolin addition to biomass with high Potassium and Chlorine such as Olive cake makes the ash compositions viable for combustion. [16] reported that Kaolin and coal fly ash which are Aluminosilicate based additives effectively captured Potassium Chloride in water slurry fed into an entrained flow reactor. [17] reported that 16% kaolin/ Kaolinite was enough to completely capture Potassium compound in empty fruit bunch during combustion process in furnace at 900°C.

AS; a biomass waste which has abundance of Aluminum and Silicon just as Kaolin, can be likened to Aluminosilicate based additive and be used to combat ash deposition problems during gasification. Up till now, there have been no report on regulating the ash deposition problem of PODC with AS. Looking into this will be worthwhile, contribute significantly to the effective thermal conversion of PODC into biofuel and aid proper management of both AS and PODC wastes. Therefore, this study intends to characterize and examine AS and PODC mix for low slagging tendency and efficient thermal process.

Materials and Methods

AS and PODC were collected from waste water treatment plant and oil palm processing factory respectively in Perak, Malaysia. Both were oven dried at 110°C for 24 hours, grinded mechanically with biomass grinder and sieve to 250 µm.

Chemical Composition Analysis:

Chemical composition analysis of the elements presents in both AS and PODC was conducted using Scanning Electron Microscopy and Energy Dispersive Spectroscopy (SEM-EDS) [Brand: Zeiss, Model: Evo LS15 VPSEM]. The scanning electron microscopy (SEM) coupled with EDS detector has the ability to determine elemental concentrations in selected spots or areas [18]. The use of SEM-EDS techniques for the detection of elemental composition have been used widely in many research activities. Samples were prepared on circular supports of 12.5 mm diameter over carbon stickers. The working distance (WD) was approximately 10 mm and the dead time (DT%) was kept at less than 30% for the experiment [19].

Thermal Decomposition:

Thermogravimetric analysis (TGA) of AS and PODC was carried out using Thermogravimetric analysis (TGA) [Brand: Perkin Elmer, Model: STA6000] in order to examine their decomposition behavior. 5.0 mg of each samples was paved uniformly in a platinum crucible and heated linearly from ambient temperature to 800°C under ambient atmosphere at a heating rate of 25°C/min. The experiment was duplicated to ensure the reproducibility of results.

Mineral Analysis:

The XRD (X-ray Diffraction) analysis was carried out to identify the crystalline compounds present in AS and PODC using Advance X-ray diffractometer (XRD) [Brand: Panalytical, Model: Xpert3 Powder]. The scan for the samples were collected from 2° to 80° (2θ scale) using Cu Kα radiation. Operating conditions are 45 kV and 45 mA. Peak identification was performed through comparison with reference standards from X'pert HighScore software package.

Ashing and ash fusion test:

2 g of AS and PODC blends in the proportion of 0% AS, 30% AS, 50% AS, 70% AS and 100% AS was ashed in a muffle furnace in accordance with National Renewable Energy Laboratory (NREL) ashing standard for biomass [20]. Deformation temperature (DT), softening temperature (ST), hemispherical temperature (HT), and flow temperature (FT) of the ashes was determined using AF700 Ash Fusion Determinator (LECO, USA). The heat treatment of ash was carried out at a heating rate of 10°C/min [21].

Result and Discussions

Proximate and Ultimate Analysis:

Table 1 shows the proximate and ultimate analysis of AS and PODC. The proximate analysis revealed the values for moisture, volatile matter, fixed carbon and ash of AS are 0.64 wt%, 3.60 wt%, 8.94 wt% and 86.72 wt% respectively. Ultimate analysis revealed that Aluminum, Carbon, Iron, Oxygen, Potassium and Silicon were the elements present with 13.50 wt %, 18.16 wt %, 2.43 wt %, 49.49 wt %, 1.46 wt % and 14.95 wt % respectively. The low percentage of volatile indicate AS is inorganic in nature and this was further proved by its low amount of carbon content. The high presence of ash content indicates its poor combustion status and increase in burning time. The values obtained was in agreement with [2] findings where 2.66% of volatile matter and 89.78% of ash content were recorded. The dominant components were found out to be Aluminum and Silicon. This resulted from the use of alum in the coagulation process. The combination of Aluminum oxide and Silicon dioxide present in AS is approximately 70% [17-18]. In PODC, the proximate analysis obtained for moisture, volatile matter, fixed carbon and ash are 2.78 wt%, 51.84 wt%, 7.13 wt% and 38.15 wt% respectively. Carbon, Oxygen, Potassium, Silicon, Calcium and Chlorine are the elements found present with 74.80 wt %, 19.76 wt %, 1.19 wt %, 1.41 wt %, 2.16 wt % and 0.67 wt % respectively. The amount of volatile matter present indicates the presence of gaseous fuels and its suitability as energy source. 74.80% Carbon content proves a good source as biomass energy. [8] reported higher volatile matter and lower ash content of PODC as 72.13% and 14.73% respectively. This is as a result of difference in location and region of the PODC used for the experiment. [24] in a similar manner reported the value of potassium and calcium in PODC to be 1.27% and 1.18% respectively while [25] gave a very close value of 74.4% for the value of organic carbon in their work.

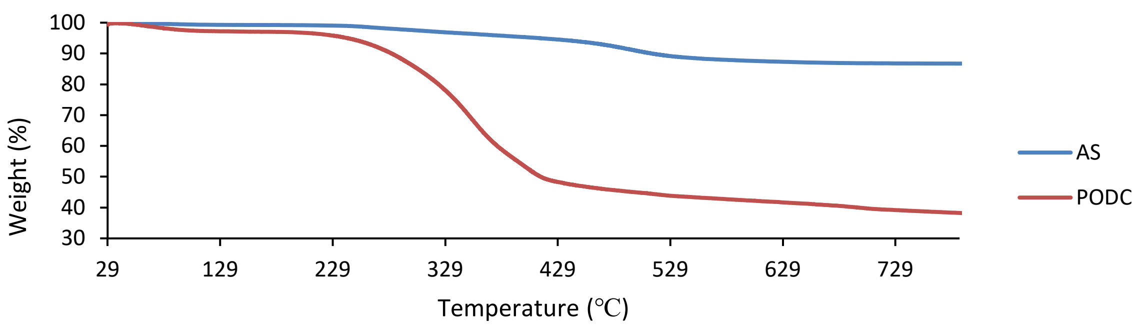
Thermal Decomposition:

Fig. 1 depicts the weight loss (TGA) and derivative weight loss (DTG) curves upon the degradation of AS and PODC at a heating rate of 25°C/min under ambient atmosphere. As observed, three distinct sections of thermal degradation were recorded for both AS and PODC and the weight loss of PODC is higher than that of AS. In AS, total weight loss of 13.29% was observed over three stages while 61.86% was observed in PODC. The first section indicates the removal of moisture present which occurred at 29-156°C for both AS and PODC. The devolatilization stage which is the second section has the most significant change in weight loss. Devolatilization occur at 156-370°C and 156-598°C for AS and PODC respectively. The differences in temperature range resulted from the degradation of hemicellulose followed by cellulose compound present in PODC. Hemicellulose was degraded first, followed by cellulose which has a long polymer of glucose with low branching structure [25]. Due to lesser volatile matter, AS was less reactive compare to PODC. The third section showed a slow degradation in weight loss as combustion occurred at 370-800°C and 485-800°C for AS and PODC respectively. The slow degradation experienced in PODC was as a result of degradation of carbonaceous material and compounds [26]. As reflected from the DTG curve peaks, it could be deduced that PODC is more reactive than AS. Similar thermal decomposition trend was reported by [27]for AS. However, a different PODC thermal decomposition report of four distinct sections of temperature 50-100°C, 100-250°C, 250-400°C and 400°C above was reported by [8].

Table 1: Proximate and Ultimate Analysis

	AS	PODC
	Proximate analysis (wt%)	
Moisture	0.64	2.78
Volatile Matter	3.60	51.84
Fixed Carbon	8.94	7.13
Ash	86.72	38.15
	Ultimate analysis (wt%)	
Aluminum	13.50	-
Carbon	18.16	74.80
Iron	2.43	-
Oxygen	49.49	19.76
Potassium	1.46	1.19
Silicon	14.95	1.41
Calcium	-	2.16
Chlorine	-	0.67

(a)



(b)

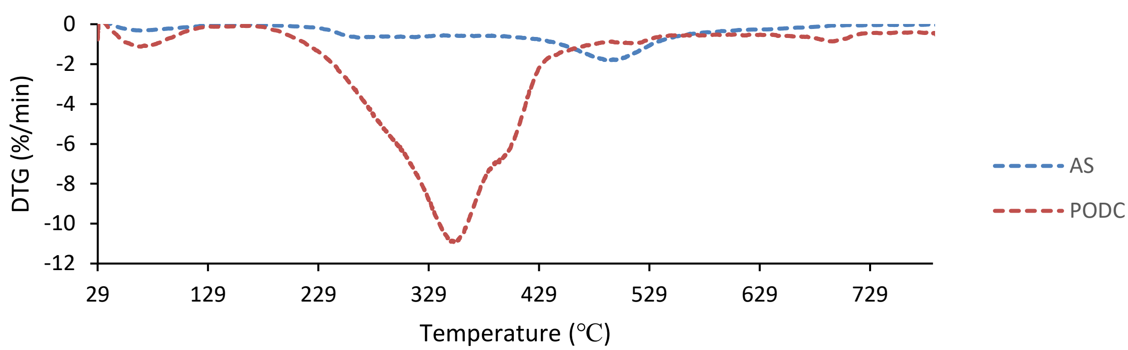


Fig. 1: Thermal decomposition curves of AS and PODC (a) TGA and (b) DTG

Mineral Composition:

The XRD patterns of raw AS and PODC are shown in Fig. 2. The patterns reveal that both AS and PODC have well defined crystalline structures as indicated by the presence of sharp peaks. Kaolinite ($Al_2H_4O_9Si_2$), Quartz/ Zeolite (SiO_2) peaks were found in the XRD structure of AS while Whewellite ($CaC_2H_2O_5$), Quartz (SiO_2) and Potassium Chloride (KCl) were found in the XRD structure of PODC. Since Aluminum Sulphate is used as coagulant in water treatment plant, the existence of Quartz/ Zeolite in AS may be due to suspended sand and clay particles in raw water [28]. Similar report about AS was given by [26]. The existence of Potassium Chloride in oil palm biomass i.e palm oil mill effluent has been reported by [29]which is basically the reason behind ash deposition.

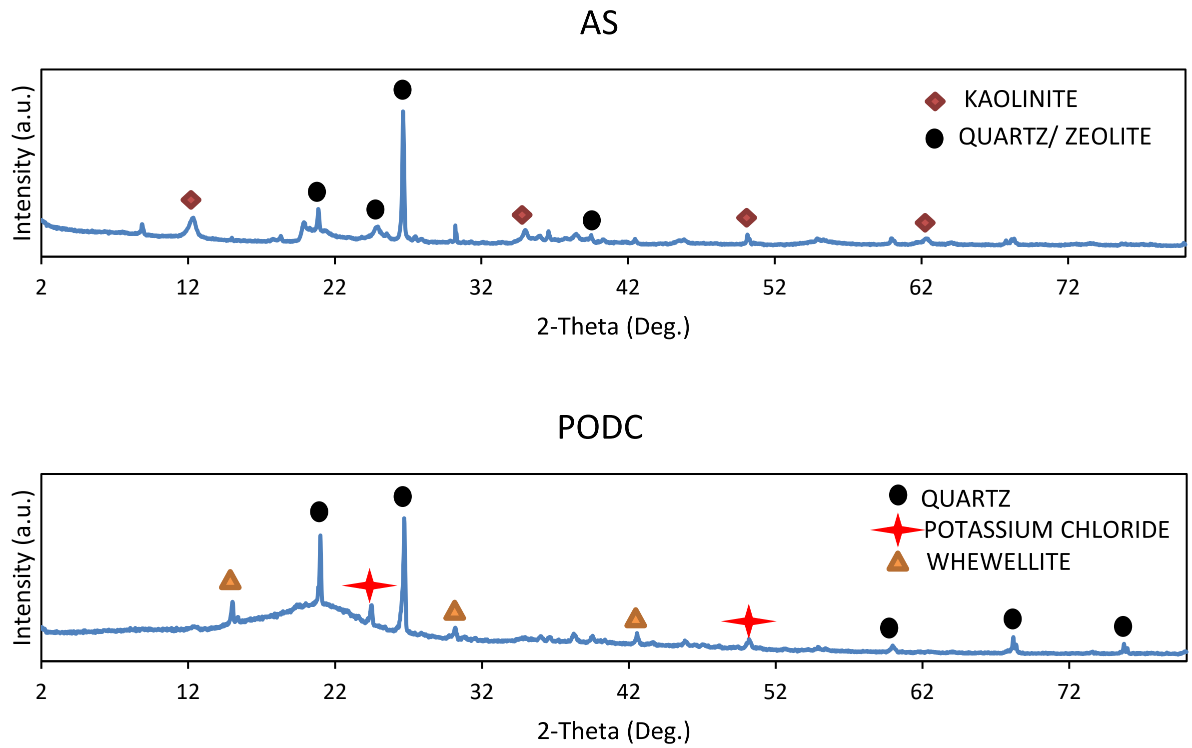


Fig. 2: XRD pattern of raw AS and PODC

Ash Fusion Test:

Table 2 shows the ash fusion temperature of AS, PODC and their blends. ST has been widely employed to predict whether a combustion process is likely to experience slagging [30]. As illustrated in Table 3, ash slagging occurs when ST is lower than 1390℃ [31]. ST for PODC and 70PODC are 1215℃ and 1280℃ respectively, indicating that the possibilities of slagging is relatively high in both samples. When the content of PODC is 50% or less in the blends, the slagging possibilities were low. The improvement in slagging when AS in the blend is 50% and above could be attributed to kaolinite in AS capturing Potassium in PODC to form high melting point Potassium Aluminum Silicate which is in agreement with [32]shown in Eq. 1 below.

$$Al_2O_3.xSiO_2 + 2KCl + H_2O \rightarrow K_2O.Al_2O_3.xSiO_2 + 2HCl_{(g)}. \quad (1)$$

Table 2: Ash fusion Temperature of AS, PODC and their blends

	DT (°C)	ST (°C)	HT (°C)	FT (°C)
PODC	1195	1215	1285	1400
AS30+70PODC	1225	1280	1350	>1400
AS50+50PODC	>1400	>1400	>1400	>1400
AS70+30PODC	>1400	>1400	>1400	>1400
AS	>1400	>1400	>1400	>1400

Table 3: Index of slagging level

Index		Slagging Level	
	Low	Medium	High
ST (°C)	>1390	1390-1260	<1260

Conclusions

In this study, AS and PODC were characterized by SEM-EDS, TGA and XRD. SEM-EDS revealed the presence of Aluminum (13.50 wt%), Potassium (1.46 wt%), Iron (2.43 wt%) and Silicon (14.95 wt%) in AS while Potassium (1.19 wt%), Silicon (1.41 wt%), Calcium (2.16 wt%) and Chlorine (0.67 wt%) in PODC. TGA revealed that fixed carbon, volatile matter and ash content in AS are 8.94 wt%, 3.60 wt% and 86.72 wt% while PODC are 7.13 wt%, 51.84 wt% and 38.15 respectively. The total weight loss of AS and PODC was 13.29% and 61.86% respectively. XRD revealed the presence of Kaolinite and Quartz/ Zeolite in AS while Whewellite, Quartz, Potassium Chloride are present in PODC. AFT revealed 1215°C and above 1400°C as the ST for PODC and AS respectively. At 50% and above of AS addition, low slagging tendency of PODC was attained. In order words, ash deposition was avoided and efficient thermal process was attained.

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