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Chaterization of *Jatropha curcas* Linn. Capsule Husk as Feedstock for Anaerobic Digestion

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Abstract

Jatropha curcas Linn. capsule husk (DH-JcL) is a residu from the manufacture of Crude Jatropha Oil. Biorefinery as part of the "four R's" was required in DH-JcL for anaerobic digestion feedstock. Analysis result and literature study was concluded that DH-JcL is a material that can be managed as biogas substrate, though the nutrient levels relatively low, and a number of other material such as volatile solid, carbohydrates, cellulose ad lignin was relatively high. The establishment of DH-JcL required two phase digestion as anaerobic microbial pretreatment and pre-acidification. The efficiency of two-phase digestion was able to increase the number of technology i.e. additives; recycling of slurry; variation in operational parameters and fixed film or biofilters utilization

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Keywords: Biogas; biorefinery; capsule husk; *Jatropha curcas* Linn.; two-phase digestion

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Nomenclature

AD	anaerobic digestion	**CJO**	crude jatropha oil
DH-JcL	dried husk *Jatropha curcas* Linn.	**PM**	partikulate matter
JcL	*Jatropha curcas* Linn.	**VFA**	volatile fatty acids
VS	volatile solid	**FFB**	fresh fruit bunches

1. Introduction

Air and water pollution, from car emissions, municipal, industrial and agricultural operations, have been growing around the world. The emission of CO_2 and other greenhouse gases (GHG) have become an important issue. *Jatropha curcas* Linn. (JcL) is one source of biodiesel which containing SO_2, CO, NO, and PM lower than petroleum-diesel [1,2]. In the other side, the facts show that the process of making Crude Jatropha Oil (CJO, the biodiesel raw materials) were not environmental friendly. There are a number of waste e.g. capsule husks (jatropha fruit coat, fruit husks, hulls, shell, fruit shell, peel, fruit encapsulation) as the subject of discussion in this paper.

The concept of the 'four R's', has been accepted generally as a useful principle for waste handling. Three R described as reduce, reuse, recycle respectively [3-10]. However, the 4th R was described in many explanation, such as rethink [3], replace [4], rot [5], reclaim [6], recover / recovery [7-9], renewable energy [10]. The principle of reuse and recycle, which called biorefinery, is recommended in the management of JcL cultivation [11-13]. The biorefinery is the zero waste process which involves waste from one process used as raw material and then transformed into other process for increasing efficiency and / or income [14,15]. CJO is only 17 % to 25 % of the dry seed weight [16,17], while the waste residue is called seed cake (*Jatropha curcas* press cake, *Jatropha curcas* defatted waste), sludge of CJO, and capsule husks [18]. Data volume of capsule husks is about 30 % to 80 % of fresh fruits weight [19,20] or 8 % to 15 % of dry weight [21].

The 4th R from the 'four R's' in references [6-10] were often called “waste to energy”. Some experts recommended capsule husk JcL as bio-briquettes / pellets [17,22,23] or as feedstock gasifier [23-25]. However, these activity conducted reuse principle only, and small portion of recycle because there is only ash as residual combustion. Most essential nutrients burned and thrown into the air, so it can’t recycle to the soil as organic fertilizer [16,26].

Recent study [3,6,7,9,10] were also recommended biological treatment, especially anaerobic digestion (AD) which declared as 'most environmentally friendly and suitable methods for the treatment of solid organic waste' [27] and supported by several experts [28,29]. AD produces biogas which stated as 'one of the most efficient and effective options among the various other alternative sources of renewable energy currently available' [30] and supported also by several experts [31-34].

Another recent study, Becker and Makkar [11,35]; Halford and Karp [36] stated that JcL husks 'was not suitable as substrates in biogas digesters because of very low digestibility'. It is also reported related to low density, high capasity buffer, and anti-nutrients (e.g. phorbol esters) which contained by JcL husks [37,38]. The literature reviews, Brittaine and NeBambi [24]; Oosterkamp [39], report JcL husks as raw material for biogas only without details explanation. Laboratory-scale researchs are reported by Lopez [40] in Nicaragua and Dhanya [41] in India only.

The studies about JcL husk as a source of biogas has been reported by several authors in Indonesia during 2010 to 2014 [42-46]. This study complements previous studies to show that JcL capsule husk is appropriate for feedstock for small scale AD / household biogas digesters, but modification or special treatment is needed.

2. Material and method

The study was conducted at the research farm of PT Bumimas Ekapersada, Bekasi, West Java, during 2010 to 2014 as part of the research data that has been reported [18,21,26,37,38,42-46]. JcL husk collected from toxic JatroMas cultivars which dried on direct sunlight, until the moisture content of approximately 5 %. Utilization of Dry Husk (DH-JcL) was aimed to provide storage efficiency so there is a continuity of biogas raw materials (JcL fruit production decrease in certain months); to increase the efficiency of entry (feeding) into the digester; and also to increase biogas production because dried samples pineapple peeling produce three times higher yields of biogas than fresh pineapple peelings [47].

Nutrient analysis on DH-JcL samples was conducted at analytical chemistry laboratory of PT Sinar Mas Agroresources and Technology Tbk., Bogor. Analysis of carbohydrates, proteins, fats, and proximat determined at Integrated Laboratory of Bogor University; Laboratory of Indonesian Livestock Research Center, Bogor; and Indonesian Center for Agricultural Post Harvest Research and Development in Bogor, West Java, Indonesia.

3. Results and discussions

3.1. The main nutrient concentrations

Kalia et al. [48]; Zhang and Zhang [49] said that the quantity and quality of biogas depends on the characteristics of organic matter feed which processed. Related to this statement, the DH-JcL nutrient analysis is determined.

Table 1. The comparation of DH-JcL nutrients content than JcL seed cake and cow dung [18].

Material	Moisture content	Ca (%)	K (%)	Mg (%)	P (%)	B ($mg \cdot kg^{-1}$)	Cu ($mg \cdot kg^{-1}$)	Fe ($mg \cdot kg^{-1}$)	Mn ($mg \cdot kg^{-1}$)	Na ($mg \cdot kg^{-1}$)	Zn ($mg \cdot kg^{-1}$)	Cl (%)	C (%)	N (%)	C / N ratio	VS (%)	Density
JcL Seed cake	6.3	0.14	0.08	0.21	0.258	6.58	4.99	140	31.3	39.8	18.1	0.47	51	3.06	17	84.5	1.20
DH-Jcl	3.49	0.03	1.05	0.03	0.012	3.99	0.27	23.5	28.9	199	1.88	0.52	48.9	1.01	49	78.6	0.59
Cow dung	1.65	1.07	1.35	0.41	0.201	8.76	11.2	29 102	983	330	199	0.24	19	0.9	21	32.8	1.01

Table 1 elucidated that the density of the DH-JcL is the lowest by 0.59. The low density of DH-JcL have the impact for this material, it will float on the substrate and clog intake channel [50], so degradation process is not optimal. Additional information about the essential nutrients was shown in Table 2.

Table 2. Nutrient content of C, N, P, and K on the DH-JcL.

DH-JcL	C (%)	N (%)	P (%)	K (%)	C / N ratio	Conclusion
[1)]A. Reference	100	5	1		20-30	
[2)]B. Analysis (%)	49	1.01	0.012	1.05	49	
C. The first alternative (Content based on A and B)	1.2	0.06	0.012			N and C <<
D. The second alternative (Content based on A and B)	49	2.45	0.49			N and P <<

Note : [1)] [51]; [2)] Table 1

Table 2 elucidated that the reference [51] required the ideal nutrient ratio C:N:P is 100:5:1. On point B, the results of analysis are listed in Table 1 consist of C = 49 %, N = 1.01 %, P = 0.012 %. Based on point A and B, point C is calculated as the first alternative and point D as the second alternative. On the conclusion column, the DH-JcL contents of N, C, and P are not appropriate as a biogas substrate because the nutrient contents is far lower than the requirements. Table 2 supports the opinion of Zhang and Zhang [49] and Parawira [51] which declaring the crop residues may lack some of the nutritional requirements than cow dung which derived from animal manure was contains large quantities of well-balanced nutrient supply. Patulasa [52] stated the substrate, which containing far lower N and P than C, was not converted perfectly elements C into CH_4. However, compared to the requirements of Malina and Pohland [53], the least number of N content is 0.4 % to 0.6 %, so the DH-JcL content still meet the requirements.

Table 2 also showed that C / N ratio of DH-JcL value of 49. C / N ratio value is higher than the requirements of

20 to 30 [54]. DH-JcL C / N ratio value was also higher than the threshold of 45 which is required by Deublein and Steinhauser [34] and Marti [55]. The high C / N ratio indicateed a nitrogen deficiency on the substrate, which resulting protein deficiency for microbial growth [54,55]. Barik and Murugan [56] and Shakya [57] said that the high C / N ratio value is indication for raw materials which producing low biogas. The high C / N ratio value produces low capacity buffer [58], the sensitive process [51,58], because the significant impact on fatty acids production and also which fatty acids are formed [59,60].

Another disadvantage of DH-JcL is the C / P ratio value. The study [54] required the C / P ratio value of 150 to 200, but Table 2 showed that the C / P ratio value was 4 083. The high number of C / P ratio value due to P content did not meet the requirements Speece [61] and / or Rahmadani [62]. Phospate is macro-mineral which required by microorganisms for microbial population growth and maintaining pH stability. Phospate deficiency will lead disruption of microbial growth, so biogas production was decreasing [52]. It is happened because the phosphate associated with ATP (adenosine triphosphate) and NADP (nicotinamide adenine dinucleotide phosphate) as an energy carrier [63] and the synthesis of nucleic acids [64]. The similar condition for the C / K ratio value of DH-JcL which not eligible too. Reference required the C / K ratio value of 40 to 100, with an optimal number of 70 [65], but Table 2 showed the value of 47.

Table 1 also showed the number of VS (volatile solids). Several researchers [66,67] stated that VS is an important parameter in forecasting the amount of methane which produced on a substrate. Goswami [68] stated that cow dung with higher ratios of VS will have greater methane productivity. It is shown in Table 1, VS DH-JcL of 78.6 % is higher than cow dung of 32.8 %. Martin [69,70] reported cow dung VS value of 29.7 % to 39.6 %, Oleszkiewicz and Poggi-Varaldo [71] stated 27.8 % to 29 %, and Singh, et al [17] discovered DH-VS JcL of 68.73 %. However, methane potential related with VS of DH-JcL, Gerradi [67] have pointed that the higher volatile solids feed to the digester the amount of volatile acids formed in the digester also large. The larger amount of volatile acids in the digester will effect to the greater impact of volatile acids on digester alkalinity and pH.

3.2. Carbohydrates, proteins, fats, and proximate analysis

Anunputtikul [72] stated organic wastes, which including domestic, industry, and agriculture wastes, can be treated using the biogas production process. The related research [51,73] confirmed that all organic material can be digested in anaerobic, as long as they contain carbohydrates, proteins, fats, cellulose, hemicelluloses, and lignin as the main components [31]. Related to this statement, analysis of carbohydrates, proteins, fats, and proximate contents was conducted as presented in Table 3 and Table 4.

Table 3. Comparation of carbohydrates, proteins, and fats content of DH-JcL than the cow dung.

Material	Carbohydrate (%)	Protein (%)	Fat (%)
DH-Jcl toksik	64.59	7.51	4.51
Cowdung*)	41.15	9.55	0.4

Note *) [18]

Table 2 shows that the largest component of the toxic categories of DH-JcL is carbohydrates. DH-JcL carbohydrate content of 156.96 % is higher than cow dung. It supports the results of Schnürer and Jarvis [74] which stated that the agricultural wastes are rich in carbohydrates. However, it has disadvantage of low buffer capacity [51,75] so it was assume that may have problem in the alkalinity of the substrate. Table 4 and Table 5 showed the approximate biogas volume, which can be formed from carbohydrates, compared to protein and fat.

Tabel 4. Theoretical methane yield of three type of organic matter.

Source	Unit	Carbohydrat (%)	Protein (%)	Fat (%)
Roediger et al. [76]	$Nm^3 \cdot kg^{-1}$ odm $_{rounded}$	0.79	0.70	1.27
Angelidaki, Ellegaard [77]	$L\ CH_4 \cdot g^{-1}\ VS_{rem}$	0.415	0.496	1.014
Berglund, Börjesson [78]	$m^3 \cdot kg^{-1}$ VS	0.38	0.53	1.0

Table 3 showed the fat content of DH-JcL potential as biogas feedstock compared with cow dung is relatively high that is 4.51 % compared to 0.4 %. Several study [34,74,76-79] stated that high fat content of raw material will increase the quantity and also the quality of biogas, as shown in Table 5.

Tabel 5. Theoretical comparation of CH_4 : CO_2 and methane content of three type of organic matter.

Source		Carbohydrat (%)	Protein (%)	Fat (%)
Roediger et al. [76]	CH_4 : CO_2	50 : 50	71 : 29	68 : 32
	Methane content	50 %	71 %	88 %
Angelidaki, Ellegaard. [77]	Methane content	50 %	50 %	70 %
Berglund, Börjesson [78]	CH_4 : CO_2	50 : 50	60 : 40	70 : 30
Krich et al [79]	CH_4 : CO_2	50 : 50	55 : 45	70 : 30
	Methane content	50 %	63 %	72 %

Carbohydrates can be divided into monosaccharide, disaccharides, oligosaccharides, and polysaccharides with different characteristics which influence on the degradation in the biogas digester. Polysaccharides can be divided into homo and hetero polysaccharides. Homo polysaccharides has major role in the biomass waste/crop residue. Table 6 lists the homo polysaccharides content of DH-JcL which compared with cow dung.

Tabel 6. The comparation of DH-JcL crude fiber content than cow dung.

Material	DH-JcL *) (A)	Cowdung**) (B)	A/B (%)	Total of 1&2 A/1&2 B or 1&2&3 A/ 1&2&3 B (%)
1)Cellulose	30.38	22.28	136.36	
2)Hemicellulose	8.50	23.55	(36.09)	
3)Lignin	20.43	12.67	161.25	
Total of 1) and 2)	38.88	45.83		(84.84)
Total of 1),2) and 3)	59.31	60.28		(98.39)

*) Mean several researchers [17,26,41]
**) Mean several researchers [41, 80,81]

Table 6 showed that the cellulose content of DH-JcL is 36 % higher than cow dung, but the hemicellulose content is 36 % of cow dung only; lignin content is 61 % higher. Cellulose is the main component of plant cell wall composer. It is found in small amount of pure state in nature but found in form of lignocellulose from lignin and hemicellulose [82]. Table 6 supports the study from Chen et al. [83] that stated the agricultural waste contain high lignin and high C / N ratio, producing low yield of biogas.

Cellulose is a long chain carbohydrate which consisting of 15 to 14 000 units of glucose molecules [84] or approximately 5 000 glucose per chain [74]. Hemicellulose is also similar to cellulose which is a polymer of sugar. However, in contrast to cellulose which is only composed of glucose, hemicellulose is composed of various types of sugar [74] which is easier hydrolyzed than cellulose [85,86]. With a higher content of hemicellulose, the cow dung has advantages compared to DH-JcL. The higher content of cellulose than hemicellulose in DH-JcL is proper because cellulose is usually the dominant structural polysaccharide of plant cell walls [87]. Table 7 showed the lignocellulose content in several residues.

Table 7. lignocellulose content in several residues

Material	Corn cobs 1)	Corn stalks2)	Rice straw3)	News papers3)	Jute fibre4)	FFB- Palm Oil5)
Cellulose (%)	45	29.80	35 to 45	40 to 55	60 to 65	44.2
Hemicellulose (%)	35	33.30	18 to 25	25 to 40	--	33.5
Lignin (%)	15	16.65	10 to 25	15 to 30	15 to 16	20.4

Note 1[88], 2[89], 3[90], 4[91], 5[92]

Table 7 showed that several residues, especially high crop residue, contain cellulose. References [93] stated cellulose-containing materials are good substrates, but their full-scale utilization encounters a number of problems, including improvement of the quality and amount of biogas produced and maintenance of the stability and high efficiency of microbial communities. The data showed 59 % of biogas in Germany produced from crop residue which containing high cellulose [31]

However, the potential of cellulose and hemicellulose (holocellulose) as a biogas substrate is inhibited and prevented by lignin. Lignin is complex non-carbohydrate macromolecules. Lignin compose matrix surrounding the cellulose and hemicellulose, which providing protective force of biomass from pests / diseases and biodegradation. Lignin is resistant highly to biological degradation, enzymatic, or chemical [85]. Several researchers [74,94,95] supported the opinion of Mussatto [85] by stating that lignin is not decomposed in the biogas digesters.

3.3. Integration of discussion

Recent study [72,74,96] recommended give longer period of fermentation due to the slow degradation of cellulose, so hydrolysis was shown to be a rate limiting step [34,97,98]. However, the application of this advice is difficult because digesters are generally designed with pH is typically maintained at more optimal conditions for methanogens to prevent the predominance of acid-forming bacteria which causing the accumulation of VFA [34,51]. Matrix of discussion 3.1 and 3.2, are listed in Table 8. Several researchers suggest to conduct pretreatment for solving the problems in Table 8, particularly points (vi) and points (vii). There are several pretreatments methods have been proposed in recent years. These are physical (mechanical and non-mechanical), chemical (acid or alkaline hydrolysis, oxidative delignification and solvent extraction), physico-chemical (ammonia fiber explosion and CO_2 and steam explosion), and biological (microbial treatment) pretreatments [99]. But this technology is relatively expensive [100] so inefficient for small scale AD / household biogas digesters.

Table 8. Matrix of discussion 3.1 and 3.2.

No	Crop residue	Obstacles	Results
i	Nutrition < [49,51]	Buffer capacity < [51,58,74,75]	Biogas production <
ii	Density < [50]	Process sensitifity [51,58]	[52,56,76-79]
iii	VS > [67]	VFA formed [59,60]	
iv	C > [74]	Microbial growth < [54,55]	
v	CN ratio > [83]		
vi	Cellulose > [88-92]		
vii	Lignin > [83]		

Montgomery and Bochmann [100] suggested anaerobic microbial pretreatment, also known as pre-acidification, two phase digestion or dark fermentation, is a simple kind of pretreatment technology in the which the first steps of AD (hydrolysis and acid production) are separated from methane production. Unger PW [101] supported that this technology is appropriate for agricultural residues containing large ammounts of slowly hydrolyzable lignocellulosics. Parawira [51] recomended for all waste with unbalanced ratio of C : N : P, such as agro-industrial residues. Pavan et al. [102] stated to the substrate with a high VFA. Colussia et al. [103] argued that this technology is suitable for substrates with high toxic, organic loading rate and pH unstable in methanogenesis.

Two-phase AD technology with modifications to apply in household biogas digesters using DH-JcL as feedstocks has been reviewed [18,21,26,37,38,42-46,50]. Therefore no single pretreatment technology is suitable for all AD systems and substrates [100], it has been also reviewed several combination techniques for enhancing biogas production [104] made from DH-JcL, i.e : use of additives [18,21,26,42,45,46]; recycling of slurry and slurry filtrate [18,37]; variation in operational parameters [37,38,42,43,44,50]; and use of fixed film / biofilters [44].

4. Conclusion

Based on analysis of the nutrients, carbohydrates, protein, fat, and crude fiber content is concluded DH-JcL does not qualify as a biogas feedstock. However for supporting application of 'four R's', DH-JcL weakness can be overcome by anaerobic microbial pretreatment, i.e two-phase digestion. This pretreatment efficiency can be improved by several combination techniques i.e additives (urea, CJO, co-digestion), recycling of slurry, variations in operational (retention time and ballasts in hydrolytic digester, concentration of soaking water in hydrolytic digester), use of fixed film / biofilters (palm fiber, glass woll, and plastic).

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