

## Increasing the Quality of Agricultural and Plantation Residues using Combination of Fiber Cracking Technology and Urea for Ruminant Feeds

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### ABSTRACT

This experiment aimed to evaluate the decrease of the fiber fraction of some agricultural and plantation residues after being treated with *Fiber Cracking Technology* (FCT) and urea. The residues included rice straw, oil palm frond (OPF), oil palm empty fruit bunch (OPEFB), cocoa pod and coffee husk. They were added with 5% urea and incubated in FCT at temperature 135°C and pressure 2.3 atm for 2.5 h. The experimental treatments were arranged as a factorial design 5 × 2, in which the first factor was various agricultural and plantation residues (rice straw, OPF, OPEFB, cocoa pod and coffee husk) and the second factor was FCT application (untreated and treated with FCT + 5% urea), performed in 4 replicates. All treatments were subjected to Van Soest analysis and *in vitro* digestibility test. The decrease of fiber fraction was confirmed with Scanning Electron Microscopy (SEM), X-Ray Diffraction (XRD) and Fourier Transform Infrared Spectroscopy (FTIR) methods. Results showed that FCT + 5% urea treatment decreased NDF, ADF, cellulose and lignin contents of all samples (P<0.05), and increased IVDMD and IVOMD in comparison to untreated samples (P<0.05). However, the treatment did not alter *in vitro* methane gas production and VFA profiles of the samples. Analyses using SEM, XRD and FTIR revealed that FCT + 5% urea treatment demolished cell wall component, decreased crystallinity index and cleaved fiber bonds. It was concluded that combination between FCT and urea 5% effectively enhances the quality of some fibrous feed materials.

**Keywords:** fiber cracking, urea, SEM, XRD, FTIR

### INTRODUCTION

Agricultural and plantation productions in Indonesia are followed by the availability of their residues so that they are relatively easy to obtain and do not require production costs. These residues include rice straw, oil palm frond, oil palm empty fruit bunch, cocoa pod and coffee husk. Such agricultural and plantation residues are promising sources of feeds especially for ruminants since they are relatively not competing with human. However, these agricultural and plantation residues have limiting factor, i.e. lignocellulosic component that difficult to digest in digestive tract of ruminants and lead to low animal productivity (Laconi & Jayanegara, 2015; Octavia *et al.*, 2017). Therefore it is important to perform certain processing techniques to the residues in order to improve their nutritive quality and in turn would contribute to productivity increase of the animals.

The processing techniques for agricultural and plantation residues consist of physical, chemical and biological techniques. Although each processing technique may be able to effectively break down lignocellulose

structure, combination among these techniques may be even more effective. Accordingly, in some previous studies, combination between high temperature, high pressure and urea have been proven to improve nutritive values of rice straw (Chaturvedi & Verma, 2013; Jayanegara *et al.*, 2017) and oil palm empty fruit bunch (Lau *et al.*, 2010; Jayanegara *et al.*, 2018). Urea is preferable than ammonia since it is safe with addition level of 1%-5% of dry matter (Van Soest, 2006), easy to use and easy to obtain. The present experiment is a continuation from those previous studies in order to elucidate deeper mechanisms regarding the nutritional value improvement of agricultural and plantation residues using high temperature, high pressure and urea combination.

This experiment aimed to evaluate the effects of Fiber Cracking Technology (FCT) and urea addition on nutritive values of rice straw, oil palm frond (OPF), oil palm empty fruit bunch (OPEFB), cocoa pod and coffee husk. Advanced instruments were used for such evaluation, i.e. Scanning Electron Microscopy (SEM), X-Ray Diffraction (XRD) and Fourier Transform Infrared (FTIR) spectroscopy methods. The agricultural and plantation residues were further evaluated biologically

by using an *in vitro* rumen fermentation method in order to confirm the effectivity of FCT + urea treatment.

## MATERIALS AND METHODS

### Sample Collection and Preparation

Samples used in the experiment were rice straw, oil palm frond (OPF), oil palm empty fruit bunch (OPEFB), cocoa pod and coffee husk. Rice straw was collected from paddy field, Dramaga, Bogor, Indonesia. The OPF and OPEFB samples were collected from PT Perkebunan Nusantara VIII, Cikalongka, Bogor, Indonesia. Cocoa pod and coffee husk were collected from Temanggung, Central Java, Indonesia. All samples were chopped, then oven-dried at 60°C for 48 h and ground by a hammer mill to pass a 5 mm screen for FCT and a 1 mm screen for Van Soest analysis, *in vitro* incubation, Scanning Electron Microscopy (SEM), X-Ray Diffraction (XRD) and Fourier Transform Infrared Spectroscopy (FTIR). The experimental treatments were arranged as a factorial design 5 × 2, in which the first factor was various agricultural and plantation residues (rice straw, OPF, OPEFB, cocoa pod and coffee husk) and the second factor was FCT application (untreated and treated with FCT + 5% urea).

This experiment used an incubator named Fiber Cracking Technology (FCT) that has a capacity of 50 L, high temperature and pressure up to 200°C and 5 atm, respectively. In the present experiment, the FCT was set at 135°C and 2.3 atm for 2.5 h. Urea treatment was applied at a concentration of 5% sample dry matter (Van Soest, 2006). Urea was solubilized in 140 ml distilled water, sprayed to 70 g sample, mixed homogeneously, and subsequently put into the FCT incubator. This experiment was conducted in four replicates. Total amount of sample inserted into the FCT incubator was 280 g.

### Chemical Composition Determination

The samples in this experiment were divided into untreated samples (control) and treated samples with urea 5% + FCT. According to Van Soest *et al.* (1991), all samples were determined for neutral detergent fiber (NDF), acid detergent fiber (ADF) and acid detergent lignin (ADL) contents. Approximately 1 g sample was boiled in 100 ml neutral detergent solution (consisted of EDTA, sodium tetraborate, SDS, monoglycoether, sodium dihydrogenphosphate and distilled water) for 1 h and the residue was rinsed with aquadest and acetone, and then oven-dried at 105°C to obtain NDF. Analysis of ADF is similar to that of NDF except that the solution used is acid detergent solution (consisted of CTAB, sulfuric acid and distilled water). The NDF and ADF contents were exclusive of residual ash. Acid detergent lignin (ADL) was measured by combining 72% sulfuric acid with ADF residue and kept for 3 h, and after that rinsed with hot water and acetone. Later, the residue was oven-dried at 105°C for 4 h and then burned in a furnace at 600°C. Cellulose was determined by difference between ADF and ADL. Analyses of NDF, ADF, cellulose, and ADL were conducted in duplicate.

### Scanning Electron Microscopy (SEM) Analysis

The samples were prepared in dry powder condition and specimen holder (stubs) that had been coated with carbon tabs. Then the surface of the specimen holder that had been coated carbon tabs was affixed to the sample as thin as possible. After that, coating process was performed by sputter coater device Quorum type Q150R ES. Coating process used gold material (gold coating), sputter current 20 mA and sputter time 60 seconds. The sample that had been coated in specimen holder was mounted in stage for SEM analysis. The sample stages were inserted into the chamber and the image was taken by SEM of the ZEISS brand with the EVOMA 10. Then the SEM image was taken by a secondary electron detector, working distance 8.0 mm, EHT 16.00 kV with 500, 1000 and 2000 magnification (Golding *et al.*, 2016).

### X-Ray Diffraction (XRD) Analysis

The samples were placed in a holder, flattened, and then inserted into the Bruker D8 Advance ECO diffractometer. The diffractometer was operated in reflection mode (40 kV, 35 mA) that used Cu-K $\alpha$  radiation ( $\lambda_1=1.54060 \text{ \AA}$  and  $\lambda_2=1.54439 \text{ \AA}$ ) and used by a step scan mode with a starting position ( $2\theta$ ) of 5.000°, step size ( $2\theta$ ) of 0.020° (76.8 s per step) and ends at ( $2\theta$ ) of 80.009° (Mishra *et al.*, 2014).

### Fourier Transform Infrared Spectroscopy (FTIR) Analysis

Pellet was made of 200 mg Potassium Bromide into mortar and inserted 2 mg sample, and then quickly mixed until homogeneous. After that, the pellet was made by a pelletizer, then stored in a dry place. The samples were subsequently inserted into the Bruker Tensor 37 Fourier Transform Infrared machine (Liu *et al.*, 2014).

### *In Vitro* Rumen Fermentation

All samples were incubated *in vitro* with rumen inoculum and McDougall buffer mixture according to Theodorou *et al.* (1994). Rumen inoculum (from fluid and solid particle) was obtained from three fistulated Ongole crossbred cattle at Biotechnology Research Center, Indonesian Academy of Sciences, Cibinong, Bogor. An amount of 0.75 g sample was inserted into a 125 ml serum bottle, and then added with 75 mL buffered rumen fluid (ratio rumen fluid:buffer was 1:4 v/v). Allocation of treatments into serum bottles as the experimental units followed a factorial randomized complete block design. Incubation was carried out in three runs (replicates) and each treatment per run was represented by two bottles. Serum bottles were then immediately sealed with butyl rubber stoppers and aluminum crimp seals to start the incubation at 39°C. Supernatant was collected for determination of volatile fatty acid (VFA) after 24 h by following the description of Jayanegara *et al.* (2016). After that, the residue was further incubated

with 75 ml pepsin-HCl 0.2 N solution for another 24 h to quantify *in vitro* dry matter digestibility (IVDMD) and *in vitro* organic matter digestibility (IVOMD), and corrected for blanks. Partial VFA consisted of C<sub>2</sub> (acetate), C<sub>3</sub> (propionate), C<sub>4</sub> (butyrate) and C<sub>5</sub> (valerate). Determination of partial VFA was conducted by using gas chromatography technique (Shimadzu GC-2010 Plus, Shimadzu Corp., Kyoto, Japan). The column used was Rtx®-Wax (Restek Corp., Bellefonte, PA, USA) with a length and an internal diameter of 30 m and 0.22 mm, respectively. The detector employed was a flame ionization detector and gas carrier used was nitrogen. The temperatures of the injector and detector were 240°C, and the oven temperature was 140°C. Methane (CH<sub>4</sub>) production was estimated by partial VFA through a stoichiometric equation (Moss *et al.*, 2000), i.e. CH<sub>4</sub> = (0.45 × C<sub>2</sub>) - (0.275 × C<sub>3</sub>) + (0.4 × C<sub>4</sub>). The accuracy of such equation had been proven to be very high through comparison between estimated methane and measured methane using an infrared methane analyzer (Jayanegara *et al.*, 2015).

### Statistical Analyses

Chemical composition data such as NDF, ADF, cellulose and ADL were descriptively tabulated. *In vitro* data were analyzed by analysis of variance (ANOVA), following a factorial randomized complete block design. The blocks were represented by a number of different rumen fluid batches and taken at different weeks. The statistical model used was as follows:

$$Y_{ij} = \mu + \alpha_i + \beta_j + \gamma_k + \varepsilon_{ijk}$$

where  $Y_{ij}$  is the observed value,  $\mu$  is the overall mean,  $\alpha_i$  is the effect of various agricultural and plantation residues,  $\beta_j$  is the effect of FCT application,  $\gamma_k$  is the block effect (replicate) and  $\varepsilon_{ijk}$  is the random residual error. Significance level was considered at  $P < 0.05$ . Significant difference between parameters was examined through a post-hoc test namely Duncan's multiple range test. Prior to ANOVA, data were checked for outlier values; any values lower than -2 or higher than 2 of their standardized residuals were categorized as outliers. SPSS software version 21.0 was used for statistical analysis.

## RESULTS

### Chemical Composition and Structure

In this study, all untreated samples had higher NDF, ADF, cellulose and ADL values than those treated with FCT and 5% urea (Table 1). The highest NDF, ADF and cellulose values were untreated OPEFB, whereas the highest ADL value was obtained by untreated cocoa pod; these treatments were samples not treated with FCT + 5% urea. The lowest NDF, ADF and ADL values were obtained in treated coffee husk, OPF and rice straw, respectively, in which these samples were treated by FCT + 5% urea. Using a Scanning Electron Microscopy (SEM) analysis at 500, 1,000 and 2,000× magnification, the cross-section of the cell wall of treated rice straw with FCT + 5% urea had been destroyed; this was

in contrast with the untreated rice straw that showed a normal cell wall structure (Figure 1). A similar pattern was observed with OPF in which its cell wall was disintegrated after FCT + 5% urea treatment (Figure 2).

The X-Ray diffractogram of untreated and treated with FCT + 5% urea of rice straw and oil palm frond is presented in Figure 3. It was revealed that percentage of crystalline structure of all samples treated with FCT + 5% urea was lower than all untreated samples (Table 2). On the contrary, the percentage of amorphous structure of the treated samples with FCT + 5% urea was turned to be higher. Using FTIR spectroscopy analysis, transmittance between untreated and treated rice straw, oil palm frond, oil palm empty fruit bunch, cocoa pod, coffee husk with FCT + 5% urea were different particularly in some wavenumber area (Figure 4). Transmittance of the treated samples were higher than those untreated samples (Table 3).

### *In Vitro* Rumen Fermentation Characteristics

Generally FCT + 5% urea treatment increased IVDMD and IVOMD of the agricultural and plantation residues ( $P < 0.05$ ; Table 4). The interaction between feed × treatment was significant for IVDMD ( $P < 0.05$ ), indicating that the effect of FCT treatment was of different magnitude for each feed. The increasing of IVDMD value of rice straw, OPF, OPEFB, cocoa pod and coffee husk after FCT + 5% urea treatment was 183, 53, 11, 73 and 123%, respectively. The FCT + 5% urea treatment did not change *in vitro* methane gas production of samples. Further, the treatment also had no significant difference on total VFA concentration and individual VFA profiles such as acetate, propionate, butyrate and valerate (Table 5).

## DISCUSSION

Fiber Cracking Technology is an incubator that able to generate high temperature and pressure up to 200°C and 5 atm, respectively (Jayanegara *et al.*, 2018). Apparently such high temperature and pressure in

Table 1. Neutral detergent fiber (NDF), acid detergent fiber (ADF), cellulose, and acid detergent lignin (ADL) contents (% dry matter) of untreated and treated agricultural and plantation residues with Fiber Cracking Technology (FCT) and 5% urea

Feed	Treatment	NDF	ADF	Cellulose	ADL
Rice straw	Untreated	75.32	56.45	30.22	8.80
	Treated	50.23	34.32	18.10	5.57
Oil palm frond	Untreated	75.00	46.32	35.13	11.53
	Treated	51.43	24.09	20.17	8.31
Oil palm empty fruit bunch	Untreated	82.14	68.75	47.21	15.35
	Treated	60.46	40.28	25.40	9.90
Cocoa pod	Untreated	70.82	65.64	31.62	27.79
	Treated	48.77	41.50	20.66	19.48
Coffee husk	Untreated	66.75	63.18	30.09	27.04
	Treated	42.73	41.21	19.69	18.04

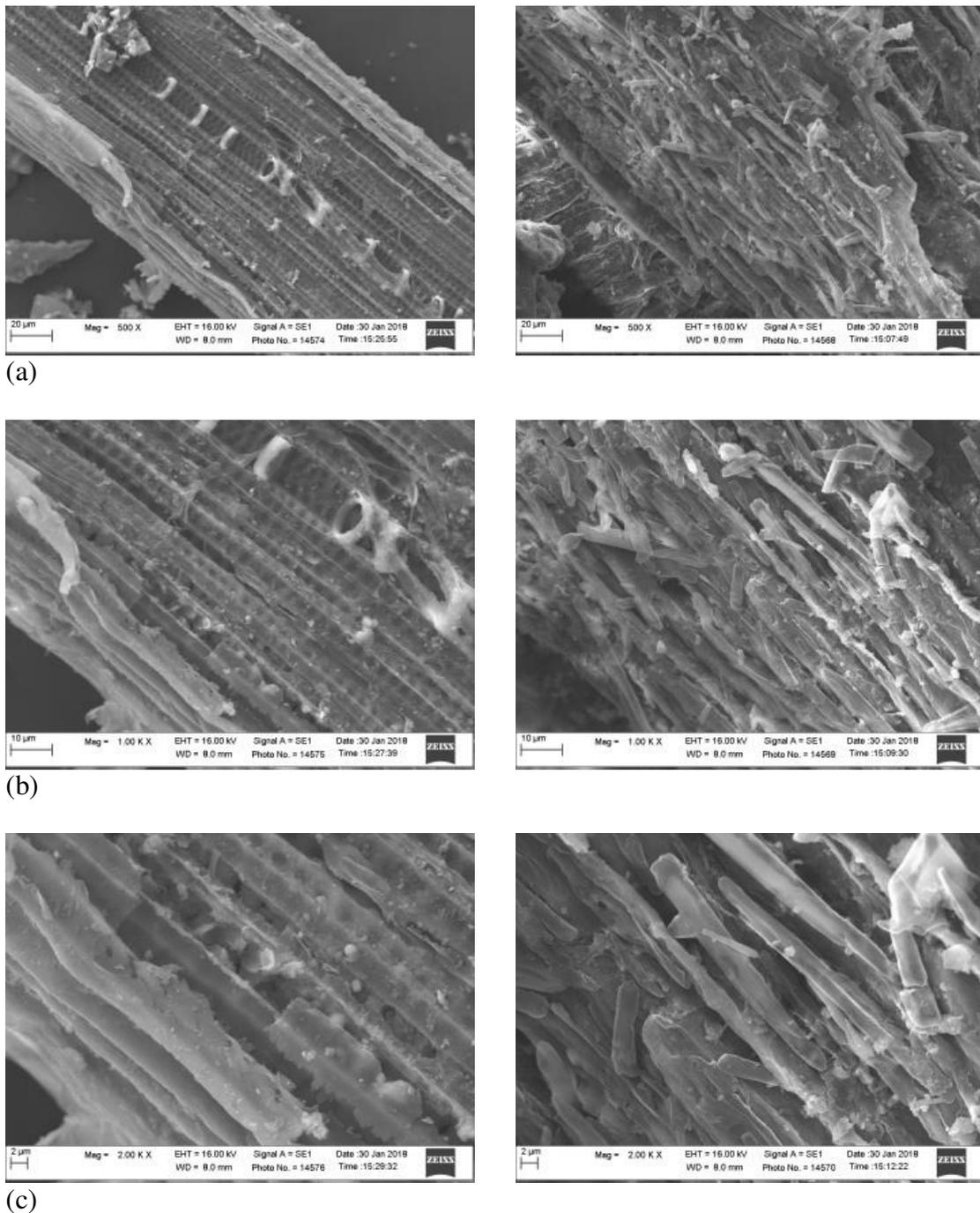


Figure 1. Scanning electron microscopy (SEM) of untreated rice straw (left) and treated rice straw with FCT + 5% urea (right) at 500× (a), 1,000× (b) and 2,000× (c) magnification.

combination with urea are able to degrade fiber fractions of agricultural and plantation residues used in the present experiment. High temperature as well as high pressure may promote acetyl group liberation from fiber structure that leads to an elevation of substrate acidity and increases fiber solubility (Thomsen *et al.*, 2014; Jayanegara *et al.*, 2018). On the other hand urea may be converted into ammonia and induces cleavage within lignocellulose structure (Shi *et al.*, 2015; Hildebrandt *et al.*, 2017). The combination of 1% urea and autoclave on rice straw increased digestibility but did not change fiber content (Jayanegara *et al.*, 2017). The combination of

5% urea of dry matter and FCT on oil palm empty fruit bunch decreased fiber fractions and increased digestibility (Jayanegara *et al.*, 2018). Therefore, the combination of FCT + 5% urea treatment improves the quality of fibrous feeds such as agricultural and plantation residues by substantially decreasing their fiber contents. The decrease of NDF value of rice straw, OPF, OPEFB, cocoa pod and coffee husk after FCT + 5% urea treatment was 33.32%, 31.43%, 26.39%, 31.14%, 35.98%, respectively. The decrease of ADF value of rice straw, OPF, OPEFB, cocoa pod and coffee husk after FCT + 5% urea treatment was 39.21%, 48%, 41.41%, 36.78%, 34.78%, respec-

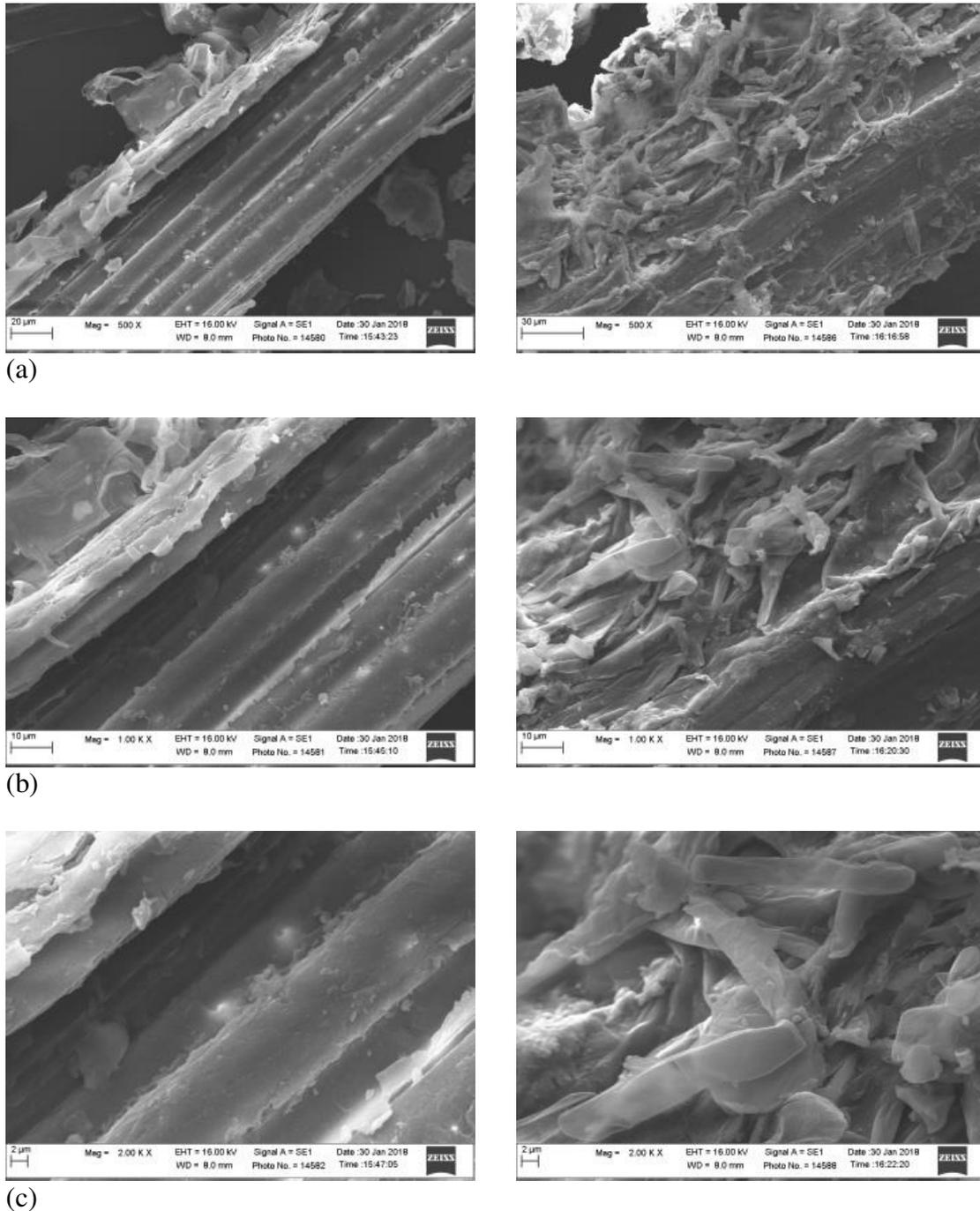


Figure 2. Scanning electron microscopy (SEM) of untreated oil palm frond (left) and treated oil palm frond with FCT + 5% urea (right) at 500 $\times$  (a), 1,000 $\times$  (b) and 2,000 $\times$  (c) magnification.

tively. The decrease of cellulose value of rice straw, OPF, OPEFB, cocoa pod and coffee husk after FCT + 5% urea treatment was 40.12%, 42.58%, 46.21%, 34.65%, 34.59%, respectively. The decrease of ADL value of rice straw, OPF, OPEFB, cocoa pod and coffee husk after FCT + 5% urea treatment was 36.69%, 27.95%, 35.54%, 29.93%, 33.31%, respectively. Combination of two or more treatments would be more efficient in order to decrease the fiber content of biomass rich in fiber component (Agbor *et al.*, 2011; Tsabitha *et al.*, 2014).

Disruption of cell wall structure of agriculture and plantation residues due to FCT + 5% urea treatment is

confirmed by SEM analysis. Microbial digestion of cell wall was promoted by the increase of ammonia (Itoh *et al.*, 1981). Hemicellulose extraction and lignin redistribution were leading to increase in cellulose accessibility that causes high digestibility of pretreated biomass. SEM images of treated rice straw showed that most of the cuticle and silica layers on the surface were broken following the treatment (Harun *et al.*, 2013). The SEM uses a beam of electrons to form a specimen image that allow the imaging and quantification of topographic features and produce an excellent representation of a three-dimensional sample (El Abed *et al.*, 2012). The

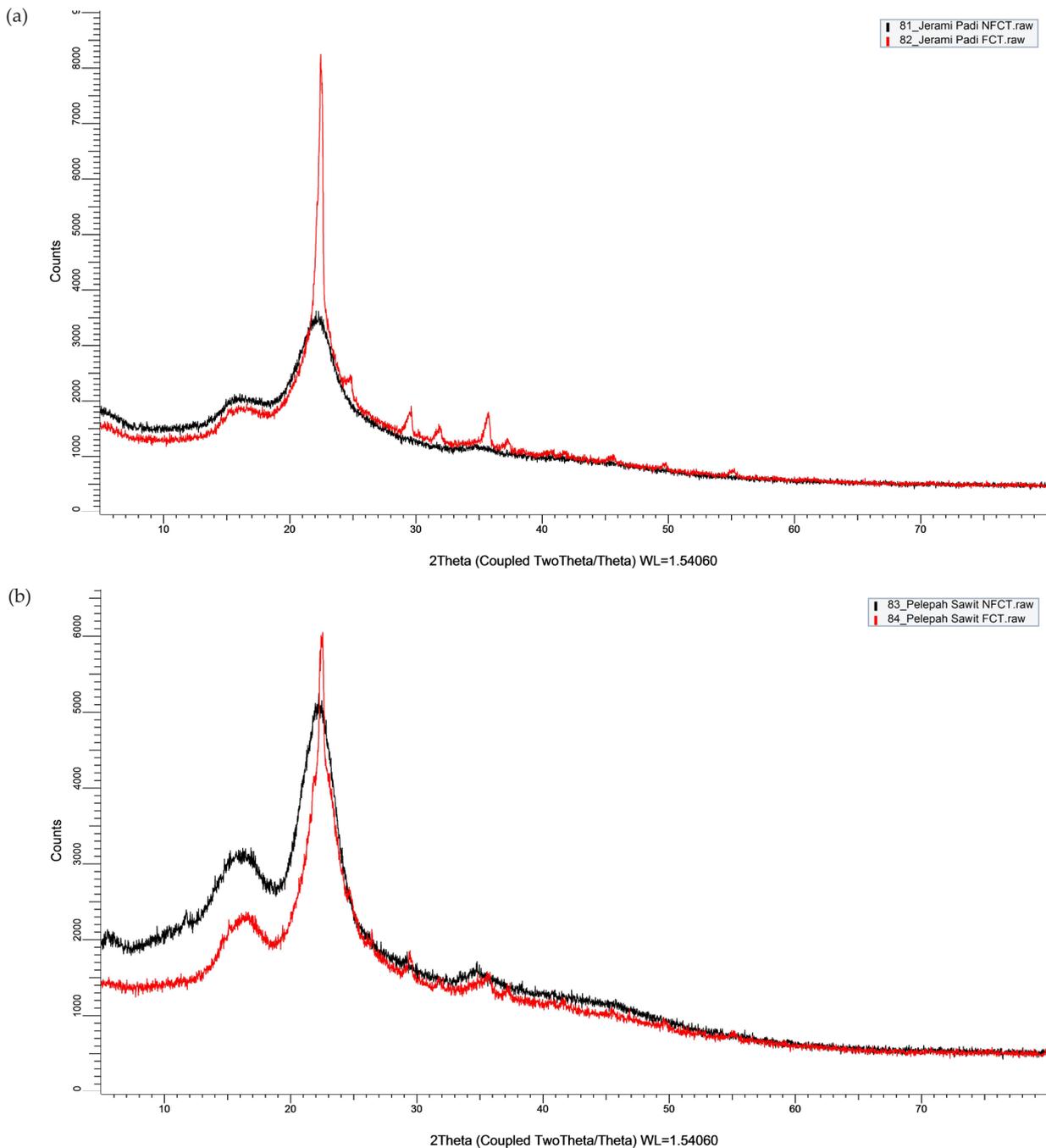


Figure 3. X-Ray diffractogram (XRD) of untreated (black color) and treated with FCT + 5% urea (red color) of rice straw (a) and oil palm frond (b).

Table 2. Percentage of crystallinity and amorphous of treatments by using X-Ray Diffraction method

Feed	Treatment	Crystallinity (%)	Amorphous (%)
Rice straw	Untreated	48.4	51.6
	Treated	44.9	55.1
Oil palm frond	Untreated	45.1	54.9
	Treated	39.4	60.6
Oil palm empty fruit bunch	Untreated	38.5	61.5
	Treated	36.9	63.1
Cocoa pod	Untreated	33.6	66.4
	Treated	30.7	69.3
Coffee husk	Untreated	43.5	56.5
	Treated	35.2	64.8

X-Ray diffraction method is used to determine the degree of crystallinity of a material with a device called a diffractometer. In this study the XRD is used to evaluate the crystallinity of cellulose present in agricultural and plantation residues before and after treatment with FCT and urea. Cellulose contains crystalline and amorphous regions that influence the enzymatic digestibility. Amorphous cellulose is hydrolyzed faster than crystalline cellulose. Ammonia liquid at 130°C for 1 h altered crystalline condition to the the disordered (amorphous) fraction and degraded cellulose (Mittal *et al.*, 2011). Lower degree of cellulose crystallinity (and higher amorphous percentage) of the materials after being treated with

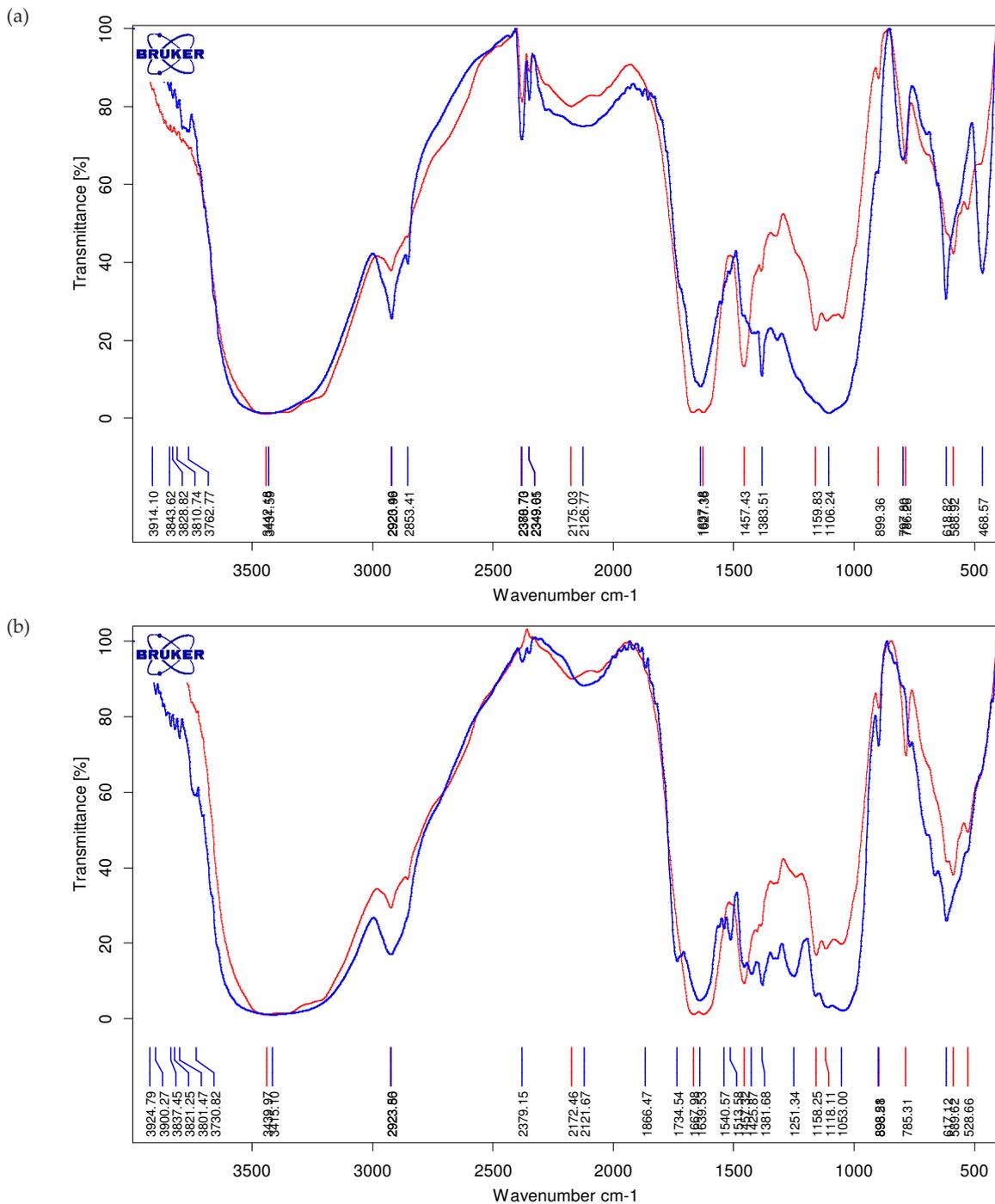


Figure 4. Fourier transform infrared spectroscopy (FTIR) of untreated (blue color) and treated with FCT + 5% urea (red color) of rice straw (a) and oil palm frond (b)

FCT + 5% urea confirms effectivity of the treatment in degrading fiber due to the breakdown of hydrogen cellulose bonding (Fatah *et al.*, 2014).

The FTIR spectroscopy may be used to identify the presence of certain functional groups in a material. The method can be used to study the chemical structure of cellulose, hemicellulose and lignin in feed as well (Adapa *et al.*, 2011). The method has been proven to be effective technique for investigating the chemical and structural changes of lignin and carbohydrate components (Liu *et al.*, 2014). Guo & Wu (2008) described

that FTIR can be used to study the characterization of hydrogen bonds present in cellulose. An increase of transmittance of samples treated with FCT + 5% urea indicates the reduction or breakdown of C-O-C bonds of lignocellulose after treatment (Fan *et al.*, 2012). All treated samples with combination of FCT + 5% urea did not show the peaks of wavenumber at 1,800 and 1,200  $\text{cm}^{-1}$ . Disappearance of peaks at approximately 1,800 and 1,200  $\text{cm}^{-1}$  shows degradation of lignocellulose complex since these peaks are regarded as carboxylic and carbonyl bonds, respectively, that part of intermolecular

Table 3. The peaks of wavenumber and bonds of untreated and treated agricultural and plantation residues with Fiber Cracking Technology (FCT) and 5% urea

Feed	Treatment	Peak wavenumber (cm <sup>-1</sup> )	Bond
Rice straw	Untreated	1106.24	C-C, C-OH, C-H ring and side group vibrations
		1383.51	In the plane C-H bending
		2126.77	C-H symmetrical stretching
	Treated	1159.83	C-O-C asymmetrical stretching
		1457.43	HCH and OCH in-plane bending vibration
		2175.03	C-H symmetrical stretching
Oil palm frond	Untreated	1053	C-C, C-OH, C-H ring and side group vibrations
	Treated	1158.25	C-O-C asymmetrical stretching
Oil palm empty fruit bunch	Untreated	1110.72	C-C, C-OH, C-H ring and side group vibrations
		1247.29	C-C plus C-O plus C=O stretch; G condensed > G etherified
	Treated	1161.52	C-O-C asymmetrical stretching
		1318.13	CH <sub>2</sub> rocking vibration at C6
Cocoa pod	Untreated	1062.31	C-C, C-OH, C-H ring and side group vibrations
	Treated	1157.97	C-O-C asymmetrical stretching
Coffee husk	Untreated	1033.87	C-C, C-OH, C-H ring and side group vibrations
	Treated	1035.86	C-C, C-OH, C-H ring and side group vibrations

Source: Fan *et al.* (2012)Table 4. *In vitro* ruminal fermentation parameters of untreated and treated agricultural and plantation residues with Fiber Cracking Technology (FCT) and 5% urea at 24 h incubation period

Feed	Treatment	IVDMD (%)	IVOMD (%)	CH <sub>4</sub> (mM)
Rice straw	Untreated	19.70 <sup>a</sup>	18.75 <sup>a</sup>	13.95
	Treated	55.72 <sup>de</sup>	61.18 <sup>d</sup>	13.17
Oil palm frond	Untreated	31.68 <sup>bc</sup>	29.65 <sup>bc</sup>	10.03
	Treated	48.47 <sup>d</sup>	51.08 <sup>d</sup>	10.31
Oil palm empty fruit bunch	Untreated	27.52 <sup>ab</sup>	22.61 <sup>ab</sup>	12.54
	Treated	30.63 <sup>bc</sup>	37.26 <sup>c</sup>	6.92
Cocoa pod	Untreated	37.53 <sup>c</sup>	22.47 <sup>ab</sup>	10.68
	Treated	64.79 <sup>e</sup>	58.31 <sup>d</sup>	6.80
Coffee husk	Untreated	25.50 <sup>ab</sup>	18.69 <sup>a</sup>	7.89
	Treated	56.78 <sup>de</sup>	56.13 <sup>d</sup>	7.89
SEM		13.69	14.80	4.28
Significance				
Feed		*	ns	ns
Treatment		*	*	ns
Feed*Treatment		*	ns	ns

Note: Means in the same column with different superscripts differ significantly (P<0.05). \*, P<0.05; ns, non significant; IVDMD, *in vitro* dry matter digestibility; IVOMD, *in vitro* organic matter digestibility; CH<sub>4</sub>, methane; SEM, standard error of mean.

ester bonds between carbohydrates and lignin complex (Abdul *et al.*, 2016).

Higher IVDMD and IVOMD of agricultural and plantation residues after being treated with FCT + 5% urea is apparently due to lignocellulose degradation during the process; the treatment is able to cleave the bonds between lignin and cellulose to result simpler forms of carbohydrates. This is confirmed by their lower NDF and ADF contents after the treatment. In addition, change of cell wall structure from crystalline to more amorphous enables rumen microbes to colonize and degrade the component more easily (Mittal *et al.*, 2011; Octavia *et al.*, 2017). Such higher digestibility of feed materials was also observed in our previous study using

OPEFB as the sample, in which digestibility was higher with increasing level of urea application (Jayanegara *et al.*, 2018). The maximum level of urea treatment in previous experiment was 6% from substrate dry matter (Van Soest, 2006). Ammonia is further utilized by rumen microbes as a substrate of microbial protein synthesis (Schwab & Broderick, 2017; Jin *et al.*, 2018) and would be used by ruminants as a protein pool for production purpose (Jayanegara & Palupi, 2010).

Methane is one of the end products of feed fermentation in the rumen and primary substrates for methanogenesis are carbon dioxide and hydrogen, performed by certain microbial groups called archaea methanogens (Patra *et al.*, 2017). Although numerically methane

Table 5. *In vitro* ruminal volatile fatty acid (VFA) profile of untreated and treated agricultural and plantation residues with Fiber Cracking Technology (FCT) and 5% urea at 24 h incubation period

Feed	Treatment	C2 (%)	C3 (%)	C4 (%)	C5 (%)	C2/C3	Total VFA (mM)
Rice straw	Untreated	42.98	24.00	15.91	2.99	1.80	69.09
	Treated	48.38	23.32	12.85	2.68	2.11	63.59
Oil palm frond	Untreated	42.55	25.33	14.65	2.65	1.68	56.03
	Treated	41.51	24.18	16.02	2.97	1.73	57.08
Oil palm empty fruit bunch	Untreated	42.53	24.51	15.89	3.21	1.76	60.93
	Treated	46.28	23.13	12.51	2.61	2.05	45.22
Cocoa pod	Untreated	35.76	24.52	14.73	3.24	1.46	60.22
	Treated	43.47	24.65	12.34	2.51	1.77	39.59
Coffee husk	Untreated	41.36	25.33	13.59	2.51	1.63	41.44
	Treated	44.45	22.87	13.07	2.87	1.94	44.20
SEM		5.37	1.83	3.02	0.53	0.33	16.58
Significance							
Feed		ns	ns	ns	ns	ns	ns
Treatment		ns	ns	ns	ns	ns	ns
Feed*Treatment		ns	ns	ns	ns	ns	ns

Note: ns, non significant; C2, acetate; C3, propionate; C4, butyrate, C5, valerate; C2/C3, the ratio of acetate to propionate; SEM, standard error of mean.

emission seems to be different among the treatments, they were not significant. Apparently a high variation of methane data is a primary factor causing such insignificant. This is related to the insignificant VFA profiles among the treatments since methane is stoichiometrically estimated from acetate, propionate and butyrate concentrations.

## CONCLUSION

Fiber Cracking Technology combined with addition of 5% urea is able to substantially decrease the fiber component in some agricultural and plantation residues. The treatment further leads to the increase of their digestibility in the rumen *in vitro*. The highest NDF decrease by the treatment was observed in coffee husk (35.98%), whereas the highest ADF and ADL decrease were found in oil palm frond and rice straw by 48% and 36.69%, respectively. The highest IVDMD and IVOMD increase due to the treatment was observed in rice straw by 183% and 226%, respectively. The breakdown of lignocellulose bonds in the residues after FCT + urea treatment is confirmed in SEM, XRD and FTIR analyses. Fiber Cracking Technology is therefore a promising technique to be applied in feed industry for ruminants.

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