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Relationship between Calorific Value and Elementary Composition of Torrefied Lignocellulosic Biomass

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Abstract: In this study, the relationship between calorific value and elementary composition of torrefied oil palm wastes (empty fruit bunches, mesocarp fiber and kernel shell) and other lignocellulosic biomass is discussed. Several correlations for calorific value vs. elementary composition for biomass were examined for their applicability to torrefied lignocellulosic biomass. One of the correlations was selected as the most appropriate for the purpose, based on average absolute error between observed and estimated calorific values. In addition, the triangle plot of carbon, hydrogen and oxygen contents in untreated and pyrolyzed biomass is proposed as an appropriate tool for discussing biomass decomposition behavior.

Key words: Lignocellulosic biomass, torrefaction, calorific value, elementary composition

INTRODUCTION

Biomass is one of the promising renewable energy sources and is utilized as solid, liquid and gas fuels. Lignocellulosic biomass wastes especially are attracting interest worldwide, because of their non-edible characteristic. Because of their availability in Malaysia, oil palm wastes are best among biomass wastes (Yusup *et al*., 2009).

In 2008, Malaysia was the second largest producer of palm oil with 17.7 million tonnes or 41% of the total world supply while Indonesia was the world largest producer of palm oil with 19.3 million tonnes of oil or 45% of the total world supply (MPOB, 2008b). In 2008, productive oil palm plantations in Malaysia covered 4.5 million hectares, a 4.3% increase from the figures for 2007 which stood at 4.3 million hectares (MPOB, 2008a). The type of biomass produced by the oil palm industry includes empty fruit bunches (EFB), mesocarp fiber, kernel shell, fronds and trunks. EFB, mesocarp fiber and kernel shell are either utilized or discarded at palm oil mills. Similarly, the rest, fronds and trunks, are either utilized or discarded at plantations. The amount of each type of biomass is summarized in Table 1. Since the current primary energy supply in Malaysia is about 70 Mtoe (million ton of oil equivalent), the total oil palm biomass energy potential of 25 Mtoe may be able to contribute considerably to decrease the consumption of fossil fuels (natural gas, coal and oil).

In order to utilize biomass wastes efficiently, the following drawbacks about biomass compared to fossil fuels must be solved properly:

- High energy consumption for collection
- Heterogeneous and uneven composition
- Low calorific value
- Raw material difficult to transport

Since it is very difficult to solve all the problems at the same time, we focus on the fourth problem in this study. There are a few options to solve this; the major ones are pelletization, liquefaction and gasification of biomass.

Table 1: Oil palm biomass and their energy potential (Yusup *et al*., 2009)

Site	Waste type	Emission rate	Plantation area or FFB received	Annual emission Million t-wet year^{-1}	Annual energy (dry base) GJx 10^{-6}	 Mtoe
Plantation	Trunk	37 t-dry ha^{-1} replantation year^{-1}	0.08 million ha replantation	10.2	51.8	1.2
	Fronds	10 t-dry ha^{-1} plantation year^{-1}	4.488 million ha replantation in 2008	154.8	704.6	16.8
Mill	EFB	20 t-wet/t-FFB (%)	90.57 million t-FFB	18.1	100.2	2.4
	Fiber	12 t-wet/t-FFB (%)	Received in	10.9	115.4	2.7
	Shell	5 t-wet/t-FFB (%)	2008	4.5	69.5	1.7
Total					1041.5	24.8

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Pelletization includes the following processes: drying, chipping, grinding and pelletizing of lignocllulosic biomass. Though pelletization is the least expensive option, there are some problems associated with it; lower heat value and quality deterioration by moisture (pellet disintegration, moss growth and bioorganic decomposition). In recent investigations, a low temperature treatment at 200 to 300°C under an inert atmosphere was found to be effective for improving the energy density and the shelf life of biomass. The treatment is called torrefaction and was reported for wood and grass biomass over the past few years (Couhert *et al*., 2009; Arias *et al*., 2008; Bridgeman *et al*., 2008; Prins *et al*., 2006a-c; Uslu *et al*., 2008). Arias *et al*. (2008) torrefied woody biomass (eucalyptus) at 240 to 280°C and found that the grindability of the biomass was improved.

Prins *et al*. (2006a) proposed a kinetic model of torrefaction (Prins *et al*., 2006b) and reported the details of torrefaction mass balance (Prins *et al*., 2006c). Some papers focused on the fuel quality (Bridgeman *et al*., 2008) and the feedstock quality for gasification (Couhert *et al*., 2009; Prins *et al*., 2006a) of the torrefied lignocellulosic biomass. Uslu *et al*. (2008) focused on comparison of torrefaction, fast pyrolysis and pelletization from the viewpoint of the international bioenergy logistics. Currently, experimental torrefaction studies are mostly conducted on woody and grass biomass; wood dusts (Uslu *et al*., 2008), beech (Couhert *et al*., 2009; Prins *et al*., 2006b, c), eucalyptus (Arias *et al*., 2008), willow (Bridgeman *et al*., 2008; Prins *et al*., 2006b, c), larch (Prins *et al*., 2006b, c), canary grass (Bridgeman *et al*., 2008). Few academic studies have been found for torrefaction of agricultural lignocellulosic wastes, such as wheat straw (Bridgeman *et al*., 2008; Prins *et al*., 2006b, c), although, they are among the promising renewable resources especially in Southern Asia (Yusup *et al*., 2009).

The calorific value of biomass and biomass-derived fuels is one of the important fuel properties, which defines the energy density of those fuels. Since the estimation of this value from the elementary composition has been recognized as an important step for performance modeling of biomass and biomass-derived fuels, many correlations have been reported (Channiwala and Parikh, 2002; Abe, 1988; RITE Tokyo Central Laboratory, 2005; Demirbas, 2001a; Selvig and Gibson, 1945; Seyler, 1938; Tilman, 1978; Demirbas *et al*., 1997). Nonetheless, no report has been found that correlates the calorific value of torrefied biomass with its elementary composition.

In this study, we focus on the calorific value and the elementary composition of torrefied oil palm wastes, such as empty fruit bunches (EFB), mesocarp fiber and kernel shell of oil palm, which are typical agricultural wastes in Malaysia. We have surveyed the published correlations of the calorific value of biomass vs. the elementary composition in order to discuss if those correlations can be applicable to torrefied biomass. For this purpose we also collected the published data set of the calorific value and the elementary composition for torrefied biomass samples (Couhert *et al*., 2009; Arias *et al*., 2008; Bridgeman *et al*., 2008; Prins *et al*., 2006a).

MATERIALS AND METHODS

Biomass samples: Three types of biomass waste were utilized in this study: empty fruit bunches (EFB), mesocarp fibers and kernel shell. These materials were collected at an oil palm plantation in Kelantan, Malaysia in September 2009. They were kept in a refrigerator maintained at temperatures below 5°C and used in this experimental work without any pretreatment.

Torrefaction experiment: Torrefaction of the biomass wastes was carried out using a horizontal tubular type reactor made of stainless steel, with a 100 mm internal diameter. A prescribed amount of biomass waste (6 g) was weighed and put in a ceramic crucible. The crucible was placed at the center of the reactor. After flushing the reactor with nitrogen, the temperature of the reactor was raised to different desired levels, i.e., 220, 250 or 300°C at a constant rate of 10 deg $\min^{-1}$ by an electric furnace surrounding the reactor. The temperature range (220 to 300°C) was chosen because selective decomposition of hemicelluloses occurs between 180 and 300°C. The reason we put the minimum temperature as 220°C is that we may not have a substantial torrefaction rate at less than 220°C. After a 30 min torrefaction, the heater was turned off and the reactor was left to cool down to the ambient temperature. The torrefied sample was then recovered, weighed and kept in an air-tight vessel till the characterization. Throughout the procedure described above, 0.5 L $\min^{-1}$ of nitrogen was flowed through the reactor. The experiment was repeated 3 times.

Measurement: For the three biomass wastes (wet) used in this study, the moisture content and the calorific value were measured. For the dried biomass wastes and the torrefied samples, the calorific value, elementary (CHNS) composition and ash content were measured. The moisture content was measured as follows. A prescribed amount of sample (3 g) was weighed in a ceramic crucible and was placed in an electric oven maintained at 105°C. After drying for 24 h, the sample was weighed every 1 h till the decrease in weight became negligibly small. The calorific value was measured using a bomb calorimeter, model C2000 series manufactured by IKA Werke. The calorific value from a bomb calorimeter is the high heat value (HHV), which includes the latent heat of the vapor emitted from the specimen. Elementary (CHNS) analysis was carried out using CHNS-932 supplied by LECO Corporation. The carbon, hydrogen, nitrogen and sulfur contents were obtained from the analytical experiment. The oxygen content was calculated by the subtraction. The ash content was measured as follows. A prescribed amount of sample (1 g) was weighed in a ceramic crucible and was placed in an electric furnace. The temperature was raised to 700°C. After 3 h, the furnace was turned off and was allowed to cool down. The crucible containing the ash was weighed.

RESULTS AND DISCUSSION

Untorrefied and torrefied samples: The biomass wastes used in this study and their physical properties are summarized in Table 2. Wahid (2007) reported figures of 19.1, 18.8 and 20.1 MJ kg^{-1} as the calorific value of EFB, mesocarp fiber and kernel shell, respectively. The rather small difference between this study and the literature is acceptable because the physical properties of biomass depend on surrounding conditions and harvesting season (Nunez-Regueira *et al*., 2001). The elementary analysis result in Table 2 show fairly close values with those reported in a previous study (Mahlia *et al*., 2001).

In this study, three types of oil palm waste were torrefied. The color of EFB and mesocarp fiber becomes darker as the torrefaction temperature increases, whereas their shape did not change significantly. Particularly EFB, torrefied at 300°C, exhibits an almost black color. A similar tendency was observed for a woody biomass in a previous study (Couhert *et al*., 2009).

Calorific and CHNS analysis: The results of calorimetry, elementary and ash analyses for the torrefied samples are listed in Table 3. In general, for all three type of biomass waste (EFB, mesocarp fiber and kernel shell), torrefaction resulted in a higher calorific value and a higher carbon content than those of untreated biomass samples (as in Table 2).

Calorific value *vs.* elementary composition: We surveyed the published correlations of the calorific value (HHV; MJ kg^{-1}) of biomass *vs.* the elementary composition in order to discuss if those correlations were applicable to torrefied biomass. For simplicity, the correlation that only includes the percentage of carbon, hydrogen and oxygen as expressed by Eq. 1 was selected.

$$\mathrm{HHV} = a\,C + b\,H + c\,O + d \qquad (1)$$

where, C, H and O are the mass percentage of carbon, hydrogen and oxygen in biomass, respectively; a, b, c and d are the constant parameter. Seven correlations shown in Table 4 have been acquired from references (Abe, 1988; RITE Tokyo Central Laboratory, 2005; Selvig and Gibson, 1945; Seyler, 1938; Tilman, 1978; Demirbas *et al*., 1997). Though the Dulong's correlation (Selvig and Gibson, 1945) includes the sulfur percentage term, it has been selected because of its historical significance. Using those selected correlations, the HHV was calculated and

Table 2: Biomass samples used and their physical properties

		Calorific value HHV MJ kg^{-1}		Elementary and ash analyses (wt.%) using dried sample						
Biomass	Moisture (%)	Wet	Dry	C	H	N	S	O	Ash	Form
EFB	57.2	10.57	17.02	45.53	5.46	0.45	0.044	43.40	5.12	Fiber of 1 mm in diameter
Mesocarp fiber	37.2	13.33	19.61	46.92	5.89	1.12	0.089	42.66	3.32	
Kernel shell	21.4	16.14	19.78	46.68	5.86	1.01	0.060	42.01	4.38	Irregular bowl-like chip of 10 mm long and 2 mm thick

Table 3: Results of calorimetry, elementary and ash analyses

			Elementary and ash analyses (wt.%)					
Biomass	Torrefaction temperature (°C)	Calorific value HHV (MJ kg^{-1})	C	H	N	S	O	Ash
EFB	220	17.17	46.75	4.68	1.27	0.12	41.42	5.75
	250	17.67	47.07	4.95	1.35	0.11	42.24	4.28
	300	20.41	49.56	4.38	1.27	0.02	43.19	1.58
Mesocarp fiber	220	19.03	46.93	5.50	1.83	0.10	43.30	2.34
	250	19.24	47.70	5.20	1.74	0.10	40.18	5.10
	300	22.17	48.60	4.87	2.14	0.09	40.03	4.26
Kernel shell	220	18.85	45.87	6.31	0.40	0.02	43.07	4.33
	250	19.07	51.89	5.71	0.47	0.01	38.50	3.42
	300	21.68	54.21	5.08	0.50	0.02	36.66	3.53

Table 4: Correlations used in this study

	Coefficients; a, b, c and d in HHV (MJ kg^{-1}) = a*C+b*H+c*O+d				Average absolute error between HHV (obs) and HHV (cal)	
Correlation	a	b	c	d	Untreated biomass	Torrefied biomass
Dulong* (a)	0.3383	1.4430	-0.1804	0.0000	0.075	0.089
Demirbas (b)	0.3350	1.4230	-0.1540	0.0000	0.047	0.058
RITE (c)	0.2698	0.0499	0.0000	6.1044	0.046	0.040
Seyler (d)	0.5190	1.6250	0.0010	-17.8700	0.100	0.104
Tilman (e)	0.4373	0.0000	0.0000	-0.3059	0.099	0.087
Tilman (modified) (e)	0.4373	0.0000	0.0000	-1.6701	0.047	0.034
Abe (f)	0.3391	1.4340	-0.0970	0.0000	0.112	0.074

(a): Selvig and Gibson (1945), (b): Demirbas *et al.* (1997), (c): RITE (2005) (d): Seyler (1938), (e): Tilman (1978), (f): Abe (1988)

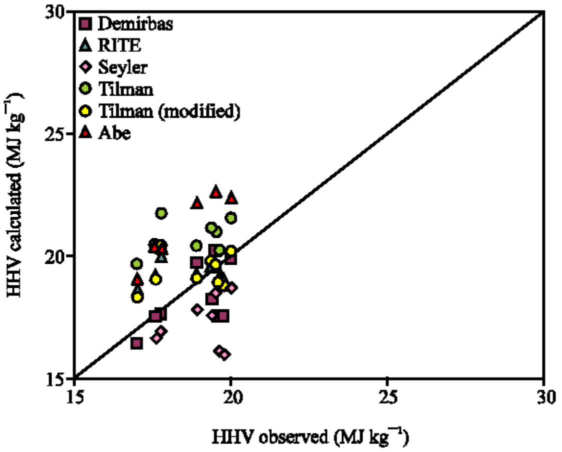

Fig. 1: Comparison between calculated and observed HHV of untreated biomass

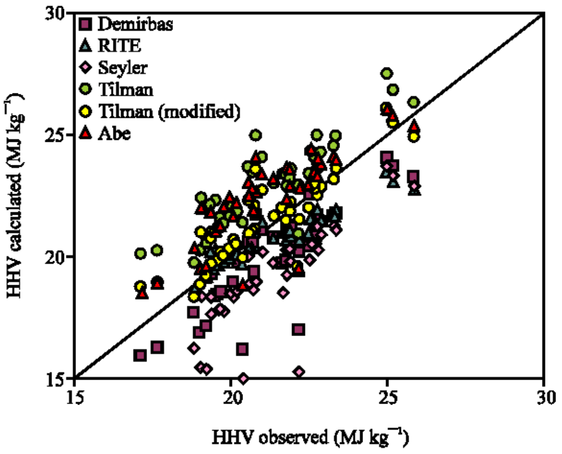

Fig. 2: Comparison between calculated and observed HHV of torrefied biomass

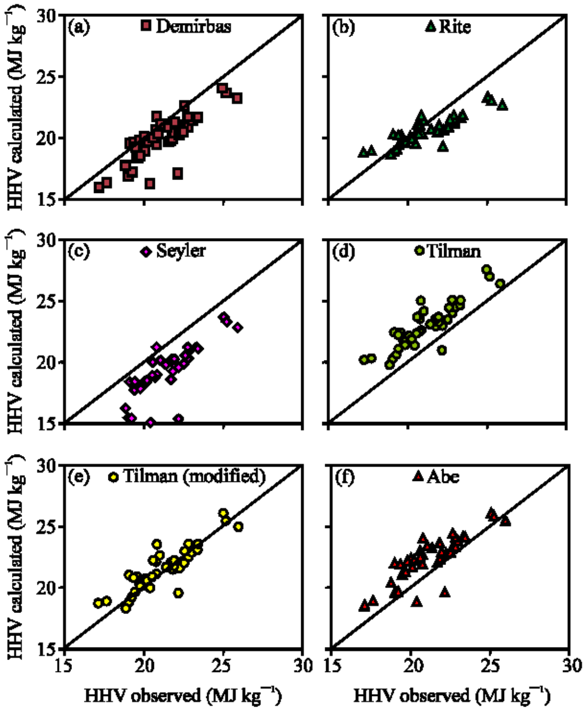

Fig. 3: (a-f) Comparison between calculated and observed HHV of torrefied biomass (separately plotted)

plotted against the corresponding experimental HHV in Fig. 1-3. The experimental data are from Table 2 and 3 and from references (Couhert *et al*., 2009; Arias *et al*., 2008; Bridgeman *et al*., 2008; Prins *et al*., 2006a). Figure 1 and 2 show the result for the untreated and torrefied biomass samples, respectively. The HHV of untreated biomass ranges from 17 to 20 MJ kg^{-1}, which is very common for lignnocellulosic biomass (Fig. 1). On the other hand, the HHV of torrefied biomass ranges from 17 to 27 MJ kg^{-1} (Fig. 2), which is higher than the values of untreated biomass.

Since it is rather difficult to clearly compare the characteristics of each correlation using Fig. 2, the result of each correlation is re-plotted separately as shown in Fig. 3a-f.

The average absolute error in each case was calculated to compare the correlations in Table 4 (Channiwala and Parikh, 2002). All the correlations can predict the HHV of torrefied biomass within a rather small range of error, even though these correlations were initially created for untreated biomass. This can be explained by the fact that torrefaction is only a partial

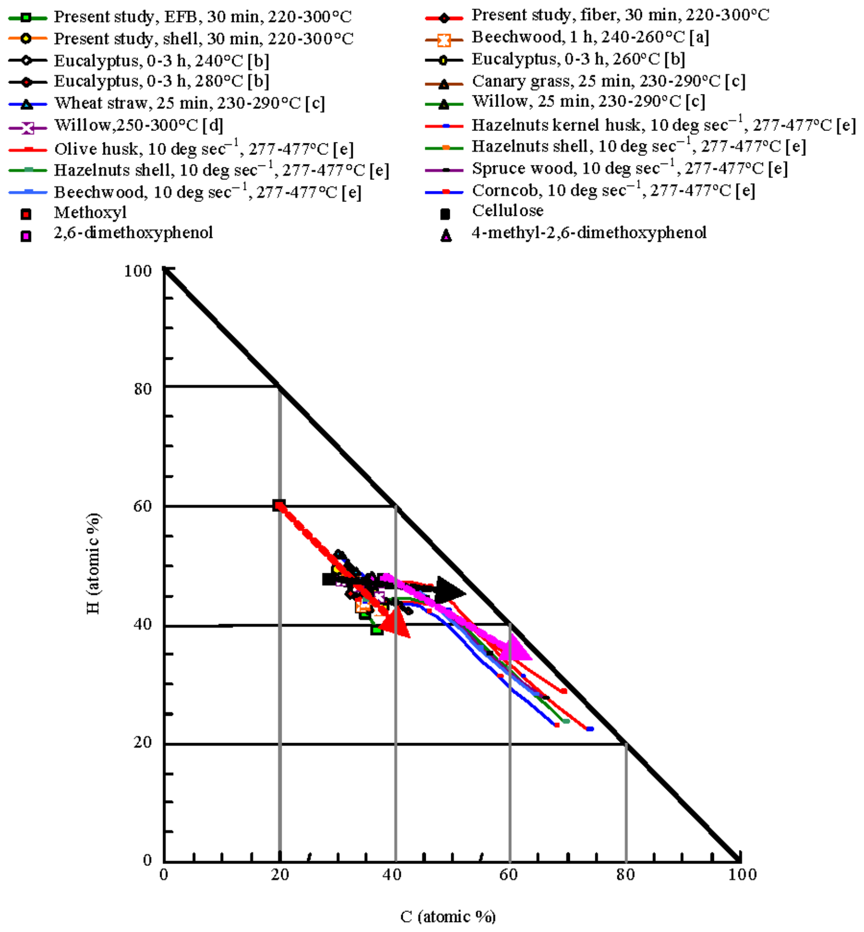

Fig. 4: Triangle plot of C, H and O for biomass and pyrolyzed biomass ([a] Couhert *et al.*, 2009; [b] Arias *et al.*, 2008; [c] Bridgeman *et al.*, 2008; [d] Prins *et al.*, 2006a; [e] Demirbas, 2001b)

pyrolysis, in which only hemicellulose decomposes (Prins *et al.*, 2006a). Obviously, the best correlation is the modified Tilman's correlation based on Fig. 3 and the average absolute error in Table 4.

It is worthwhile to mention the following two things. First, the modified Tilman's correlation only considers two terms, *i.e.*, the carbon term and the constant, whereas all other correlations have three terms as listed in Table 4. Second, the modified Tilman's correlation also gives a good estimation for untreated biomass as shown in Fig. 1.

Composition change during torrefaction: The change in elementary composition has often been expressed using the van Krevelen chart, even for torrefaction (Bridgeman *et al.*, 2008; Prins *et al.*, 2006a). In this study, we propose to use the triangle diagram, which is useful when we discuss removal or mixing of three components substances. For example, when we mix two substances consisting of three components, the resulting mixed substance must be on the straight line between the two substances before the mixing. When we remove a three-component substance from another three-component substance, the resulting substance must be on the straight line extrapolated from the two substances.

Based on the idea described above, the elementary composition of untreated and torrefied biomass is plotted in Fig. 4 using our data in Table 2 and 3 and the data from references (Couhert *et al.*, 2009; Arias *et al.*, 2008; Bridgeman *et al.*, 2008; Prins *et al.*, 2006a; Demirbas, 2001b). For more intensive discussion, the decomposition range was expanded from torrefaction (Couhert *et al.*, 2009; Arias *et al.*, 2008; Bridgeman *et al.*, 2008; Prins *et al.*, 2006a) to pyrolysis at higher temperatures (Demirbas, 2001b).

In general, the decomposition process of biomass under inert atmosphere proceeds in three steps; decomposition of hemicellulose (180-300°C), cellulose (240-400°C) and lignin (280-550°C) (Jyodai, 1993).

Torrefaction is the first step, in which the methoxyl groups of hemicellulose decompose (Demirbas, 2000). The composition of methoxyl group is shown by the red box at C = 20 and H = 60 in Fig. 4. When methoxyl groups are removed by torrefaction, the composition will move along with the red dotted arrow. The experimental data in Fig. 4 proves the prediction described above. In the second step, the cellulose decomposition, cellulose is decomposed into methyl glyoxal, acetal, propylene glycol and isopropyl alcohol successively (Demirbas, 2000) and removed from the solid phase. In this case, the composition will move along with the black dotted arrow. This is also proved by the experimental data plotted in Fig. 4. In the third step, the lignin decomposition, lignin is decomposed into char and volatile phenols; the major components are 2, 6-dimethoxyphenol and 4-methyl-2,6-dimethoxyphenol (Balat *et al*., 2009; Demirbas, 2001c, 2007, 2009). In this step, the composition moves along with the pink dotted line. This is also proved by the experimental data in Fig. 4.

CONCLUSION

In this study, torrefaction of empty fruit bunches (EFB), mesocarp fiber and kernel shell of oil palm, which are typical agricultural wastes in Malaysia, was experimentally conducted. The relationship between calorific value and elementary composition of the torrefied oil palm wastes and other lignocellulosic biomass was discussed. From several correlations for estimating the calorific value from the elementary composition of biomass, the most appropriate one, the modified Tilman's correlation, was selected. The triangle plot of carbon, hydrogen and oxygen contents in untreated and pyrolyzed biomass is proposed as an appropriate tool for discussing biomass decomposition behavior.

The result and discussion in this study will be helpful for those who will try to utilize oil palm wastes as a solid fuel or as a raw material for further conversion.

In a real torrefaction reactor, the atmosphere cannot be purely inert throughout the reactor because some types of gas, such as H_2O, CO_2 and CO, are formed by torrefaction and this changes the gas composition in the reactor, specifically at the downstream section. In this sense, further experimental work is required under a mixture of N_2, H_2O, CO_2 and CO.

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