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## **Thermal Behavior and Dust Explosion Characteristics of Spent Coffee Grounds and Jatropha as Biodiesel Feedstock**

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### **Abstract**

This work examined the minimum explosion limit (MEL), minimum ignition energy (MIE), cohesion, dispersibility, decomposition temperature ( $T_d$ ) and burning rates of spent coffee ground (SCG), jatropha kernel (JK) and jatropha shell (JS) were studied. The MIE values of oily SCG containing 21.3 wt% and oil-extracted SCG were 35 and 120 g m<sup>-3</sup>, respectively. Moreover, cohesion of oily SCG and oil-extracted SCG were high level and low level, respectively. It was found that MIE of oily SCG containing 21.3wt% of oil was low although high cohesion. While oil-extracted jatropha kernels and shells had MEL values of 45 and 110 g m<sup>-3</sup>, respectively. However, Oily JK containing 60.7 wt% of oil was not exploded reason for high cohesion and no form dust cloud. The MIE values of untreated SCG, oil-extracted SCG, oil-extracted JK and JS were found to be >3000, >3000, 1515, and >3000 mJ, respectively. These biomasses were needed high energy ignition for explosion. Burning rates of JK and JS were 0.21 and 0.04 mm s<sup>-1</sup>, respectively, these values were very slow compared with cellulose used as a reference materials was 0.67 mm s<sup>-1</sup>. Besides SCG were not capable of ignition. The  $T_d$  of both untreated SCG and oil-extracted SCG were 240 and 241 °C while the  $T_d$  of untreated JK (60.7wt% oil), oil-extracted JK, and JS were 195, 189, and 233 °C indicating that the ignition temperature is influenced by oil content. Consequently, the results demonstrate that oily

solid biomasses such as SCG and jatropha are associated with a high risk of fire, dust explosion, and related incident.

## **Introduction**

In recent years, the utilization of biomass to produce carbon-neutral substitutes for the fossil fuels that lead to global warming has become of interest. Various biomass fuels, such as biodiesel, bioethanol, refuse-derived fuel (RDF), and refuse paper plastic fuel (RPF) have been studied by many researchers [1-2] and there are numerous reports concerning the explosive and flammability characteristics of biomass fuels [3-4].

Japan also imports more than 450,000 t of coffee each year, ranking 4th in the world in term of coffee consumption, and thus generates significant quantities of waste coffee ground. The present study focused on fuels derived from spent coffee ground (SCG,) in which approximately 20 wt% of the original fatty oil content remains [5-7]. *Jatropha curcas*, which has seeds containing more than 60 wt.% oil [8], was selected as a model inedible oil-bases biomass.

In our previous study, we investigated the properties of biodiesel fuels derived from inedible oils from SCG and jatropha kernel (JK) combined with various alcohols [9]. That work included an evaluation of the thermal and oxidation stabilities of these fuels, based on analysis of their combustion and storage behaviors.

Although biomass fuels have significant potential, processing the raw materials used for these fuels entails the risk of fires or dust explosions, because these materials are processed in a powdered form.

A dust explosion requires three conditions to be present simultaneously; there must be a combustible material, an ignition source and sufficient oxygen [10]. The work reported herein was meant to evaluate the combustion behavior and associated risks of accidental dust explosions associated with SCG and jatropha biomasses. To this end, the minimum explosion limit (MEL) concentrations, minimum ignition energy (MIE), decomposition temperature ( $T_d$ ), and burning rates of these oily biomass powders were determined.

Moreover, cohesion and dispersibility of oily SCG and oil-extracted SCG were measured by powder tester in order to evaluate dust explosion effect on oil contents, cohesion, and dispersibility.

The MEL and MIE data were examined as a means of evaluating the effects of oil content in the SCG and jatropha materials, as well as the influence of either air drying or absolute drying. The presence or absence of oil was thus assessed as to its effect on the risks of dust explosion.

## Experimental

### Materials

The SCG were supplied from university coffee shop, while jatropa was obtained from Thailand. The n-hexane, as solvent to extract oil from the SCG and jatropa kernels was purchased from Wako Co. Ltd. Cellulose Avicel PH-101 for use as a reference material was purchased from Sigma-Aldrich.

SCG was dried at 80 °C for 3 days. Jatropa seed were descaled and the kernels were milled by mortar. Following these preparation step, 500 g samples of the dried SCG or milled jatropa kernel were leached six times, by 1 L of n-hexane for 1 h period at room temperature. The extraction residues were subsequently filtered and the solvent was removed by evaporation. The removed oil contents of the SCG and jatropa were determined to be approximately 20 and 60 wt%, respectively.

### Physicochemical characterization

The elemental compositions of samples in terms of carbon, hydrogen, and nitrogen content were determined by a CHN analyzer (PerkinElmer-2400 II) based on the JIS M8819 standard. The moisture and ash contents were calculated from the dry weights of samples heated at 105 and 600 °C, respectively, based on the JIS M8812 standard. The total calorific values of samples were determined using an auto-calculating bomb calorimeter (Shimazu, CA-4PJ) based on the JIS M 8814 standard. Detail oil contents were determined by Soxlet extraction with n-hexane for 8 h.

### Evaluation of dust explosion hazards

During this work, both the MEL and MIE values were determined by 1.2-L Hartmann tube apparatus (Seishin Enterprises, PIE-1200). The MEL measurement conditions consisted of: electrode gap of 5 mm, compressed air at 0.075 MPa, charging voltage of 1000 V, and ignition delay time of 0.1 s. Using these test parameters, the MEL of a standard reference material composed of Lycopodium was found to be  $40 \pm 5 \text{ g m}^{-3}$ . During MIE measurements, the discharge energy was obtained by adjusting the ohmic value of the circuit with discharge time.

Explosion hazards may be placed into three levels based on the MEL value: high ( $<40 \text{ g m}^{-3}$ ), moderate ( $40\text{--}100 \text{ g m}^{-3}$ ), and low ( $>100 \text{ g m}^{-3}$ ). Similarly, there are three levels based on MIE value: high (1–10 mJ), moderate (10–100 mJ), and low (100–1000 mJ). Energy value

calculations were performed using the following equation, according to the EN 13821 standard.

$$E_S = 10^{(\log E_2 - \frac{I[E_2](\log E_2 - \log E_1)}{(NI+I)[E_2]+1})}$$

Here,  $E_1$  is the highest energy at which ignition does not occur,  $E_2$  is the lowest energy at which ignition is observed,  $NI$  is the number of non-ignitions observed at  $E_2$ , and  $I$  is the number of ignitions observed at  $E_2$ .

### Evaluation of flow properties of biomass powders

Cohesion as the flowability value and dispersibility as the floodability values of SCG were evaluated by powder tester measurement (Powder tester PT-X, HOSOKAWA MICRON CO.). Flowability and floodability were classified following the evaluation method which was reported by R.L. Carr et.al. [11].

### Evaluation of flammability

Burning rate tests were performed based on the United Nations Transport of Dangerous Goods Method N.1: Test method for readily combustible solid [12]. The thermal behaviors of the oily biomasses were also examined based on TG-DTA measurements (Seiko Instruments, TG/DTA 6200) over the temperature range of 30–700 °C at a rate of temperature increase of 10 °C min<sup>-1</sup> under an air flow of 100 mL min<sup>-1</sup>.

## **Results and discussion**

### Particle size distribution of SCG

Particle size of espresso SCG and drip SCG were measured by using sieves. A result of particle size distribution was shown particle size distribution of espresso SCG was smaller compared with drip SCG. Consequently, espresso SCG was selected for evaluation of dust explosion tests.

Table 1. Particle size distribution of espresso SCG and drip SCG of air dry state.

Particle size ( $\mu\text{m}$ )	Particle size distribution (%)	
	Espresso SCG	Drip SCG
over 590	0.0	9.7
590-350	63.0	61.1
350-250	15.2	12.9
250-210	6.3	4.4
210-177	3.8	2.8
177-105	2.9	2.3
under 105	2.3	1.8
loss	6.5	4.9

### Physicochemical characteristics

The CHN elemental compositions, total calorific values, oil contents and ignition temperature of samples are summarized in Table 2. The total calorific values of these biomasses were found to be higher than that of cellulose, and thus they offer better energy efficiency than woody fuels.

Table 2. Physicochemical characterization of SCG and jatropha

Factor		Spent coffee ground		Jatrop			Cellulo
		Espress	Oil	ha			se
		Untreat	extracted	Untre	Oil	Untre	
		ed		ated	extracted	ated	
Element (dry %)	C	55.86	49.9	60.86	44.14	41.16	41.63
	H	7.02	6.19	9.73	7.65	5.43	6.73
	N	2.33	2.53	3.55	6.9	1.21	0.07
Total calorific value (MJ/kg)		23.56	20.85	29.74	20.38	15.96	16.68
Content of oil (wt.%)		21.27	-	60.7	-	1.86	-

Ash (%)	2.17	2.47	4.56	9.61	8.81	-
Moisture content (wt%)	3.67	7.08	5.99	5.06	10.3	4.18

#### Minimum explosion limit concentration

Figure 2 shows the MEL values of the SCG with regard to the oil content, the particle size, and the state of drying state. The results show that the MEL values for the untreated SCG and for the absolutely dry, oil-extracted SCG with particles sizes in the range of 75–105  $\mu\text{m}$  were 120 and 35  $\text{g m}^{-3}$ , respectively, demonstrating that the explosion hazard presented by SCG increases with oil extraction. Because the MEL value of the thoroughly dried SCG was lower than that of the air-dried material, the explosion hazard is evidently increased with elevated levels of drying. The MEL of the SCG corresponded with powdered milk (60  $\text{g m}^{-3}$ ) and potato starch (125  $\text{g m}^{-3}$ ) [13].

Figure 3 also shows the MEL values of the JK and JS. MEL of oil-extracted JK and shells with particle sizes of 75–105  $\mu\text{m}$  were 45  $\text{g m}^{-3}$  and 130, respectively at absolute dry state. Although not shown in Figure 3. JK containing 60 wt.% oil is not explosive because may be not form the dust cloud by the cohesion occurred in oily particles.

#### Minimum ignition energy

Table 3 shows the MIE values of samples in 125–177  $\mu\text{m}$  particle size. These results indicate that the MIE of SCG, oil-extracted SCG, and JS were all over 3000 mJ, meaning that these powders present low ignition risks. The MIE of the oil-extracted JK was 1515 mJ, indicating that this material presents a higher risk compared with the other powders. The energy required to initiate an explosion with the SCG materials was very high, but dust explosions were still possible at very low dust concentrations. The material that presents the greatest risk of a dust explosion is oil-extracted JK with particles sizes below 125  $\mu\text{m}$ .

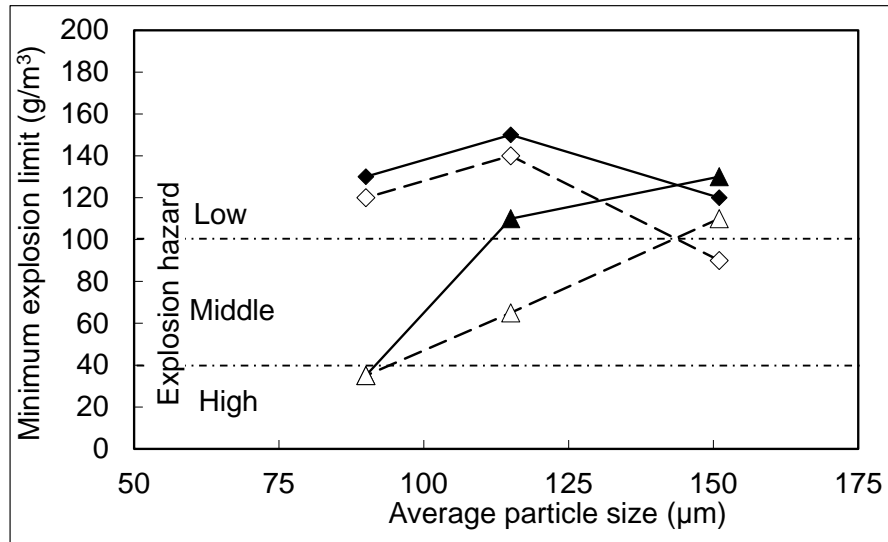


Figure 2. Minimum explosion limit concentration of spent espresso coffee grounds. Closed and open symbols were air dry state and absolute dry state, respectively. Diamond shape and triangle were oily SCG and oil-extracted SCG, respectively.

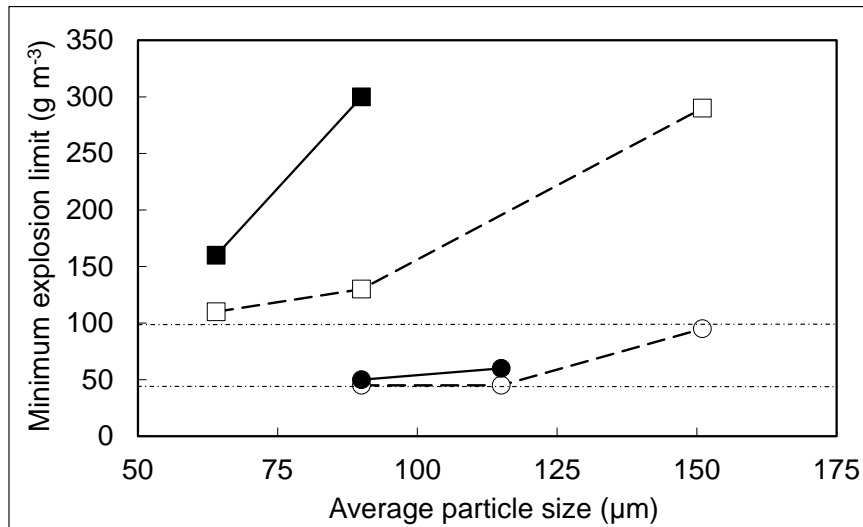


Figure 3. Minimum explosion limit concentration of jatropha kernel and jatropha shell. Closed and open symbols were air dry state and absolute dry state, respectively. Square and circle were Jatropha shell and oil-extracted jatropha kernel, respectively.

Table 3. MIE of biomasses samples under absolute dry state

Sample	Treated	Particle size ( $\mu\text{m}$ )	MIE (mJ)
SCG		125–177	3000<
SCG	oil-extracted	125–177	3000<
Jatropha kernel	oil-extracted	125–177	1515
Jatropha shell		125–177	3000<
Cellulose		ca. 50	407

### Cohesion and dispersibility of SCG

Cohesion and dispersibility of SCG and oil-extracted SCG of 125-177  $\mu\text{m}$  particle size was evaluated by powder tester. Dispersibility index of oily SCG and oil-extracted SCG were 8.0 and 12.0, respectively. This index is evaluate 25 levels, the high index point is, the more floodability is show. Beside, cohesion index of oily SCG and oil-extracted SCG were 7.0 and 14.5, respectively. This index is evaluate 15 levels, the high index point is, the more flowability is show. Dispersibility index of oily SCG and oil-extracted SCG were similar. Oil contents had no effect on dispersibility.

Cohesion index of oily SCG was very low compared with oil-extracted SCG. However, MEL concentration of oily SCG was low in spite of high cohesion. It was considered that oil contents was promoted to dust explosion even apparent particle size is large reason by high cohesion. Especially, it is necessary to attention when an ignition source energy is high.

### Flammability

The burning rate values are shown in Table 4. Powder is classified in easily combustible material when rate of burning is more than  $2.2 \text{ mm}^2/\text{s}$  in standard. These results indicate that the oily biomasses either exhibited low burning rates or self-extinguishing almost immediately after being ignited. Despite these results, however, it remains necessary to deal carefully with oily biomasses because they still present a combustion risk.

The thermal behaviors of the oily biomasses were observed by TG-DTA measurements. Table 4 also summarizes the  $T_d$  values, exothermic peak top temperatures ( $T_{\text{exo}}$ ), and peak ranges of the various materials. The  $T_d$  values of all the oily materials were lower than that of cellulose. In the case of the SCG, the  $T_d$  values of all versions tested were nearly identical regardless of the presence or absence of oil. For JK the  $T_d$  depended on the oil content, because the  $T_d$  were



higher in order of oil-extracted (189 °C), untreated ones (195 °C), only oil (202 °C).

$T_{\text{exo}}$  of SCG was lower by 4 compared with oil-extracted ones.  $T_{\text{exo}}$  of JK was higher by 76 °C compared with oil-extracted ones. It is considered that oil contents is related although it is not yet clear. However, oily biomasses have risk for combustion accident, whereas burning rate is low.

Table 4. Thermal decomposition measured by TG/DTA and burning rate test

Sample	Treatment	UN burning rate test (mm/s)	$T_d$ (°C)	$T_{\text{exo}}$ Peak Top (°C)	Peak Range (°C)
Cellulose		0.67	294	352	330–390
Espresso SCG		N.D.	240	510	475–530
Espresso SCG	oil-extracted	N.D.	241	514	475–540
Espresso oil		-	211	365	305–390
Jatropha kernel		0.21	195	422	420–440
Jatropha kernel	oil-extracted	N.D.	189	346	290–400
Jatropha kernel oil		-	202	302	219–335
Jatropha shell		0.04	233	455	400–520

N.D. No detected by self-extinguishing

## Conclusions

The MEL values of the SCG before oil extraction and the completely dry SCG after oil extraction (75–105  $\mu\text{m}$  particles) were 120 and 35  $\text{g m}^{-3}$ , respectively. MEL concentration of oily SCG was low value in spite of high cohesion. It was considered that oil contents was promoted to dust explosion even apparent particle size is large reason by high cohesion. The MIE of oily SCG, oil-extracted SCG, and oil-extracted jatropha kernels and shells were >3000 mJ, >3000 mJ, 1515 mJ, and >3000 mJ, respectively, while the MIE of cellulose (<50  $\mu\text{m}$ ) was 407 mJ. The burning rates of JK containing 60.7 wt% of oil and JS were 0.21 and 0.04  $\text{mm s}^{-1}$ , respectively, and that of cellulose was 0.67  $\text{mm s}^{-1}$ . However, could not be ignited. These results reinforce the necessity of carefully studying the explosion characteristics and combustion behavior of oily

powders.

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