

# Recovery of Oil from Spent Coffee Grounds by Solvent Extraction for Biodiesel Production

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## 1. Introduction

Coffee is the second most consumed beverage in the world after water [1] and a huge amount of spent coffee grounds (SCG) is generated after brewing coffee. The amount of SCG generated from the instant coffee industry in the world was estimated about 6 million tons per year [2] and various approaches to utilize SCG have been developed, such as fertilizer, wood powder, absorbent and so on. In spite of these applications, most of the SCG are still discarded as waste and the effective utilization of SCG is being studied. Although the utilization of the SCG in the biofuel production was expected as a promising application because of its great sugar content and lipid content, the production method has not been fully studied.

This study aims to apply the solvent extraction technique to the recovery of oil from the SCG for biodiesel production. Then, the overall yield of the oil recovered from SCG was determined and effects of the solvents and extraction temperature on the recovered oil were studied.

## 2. Experimental

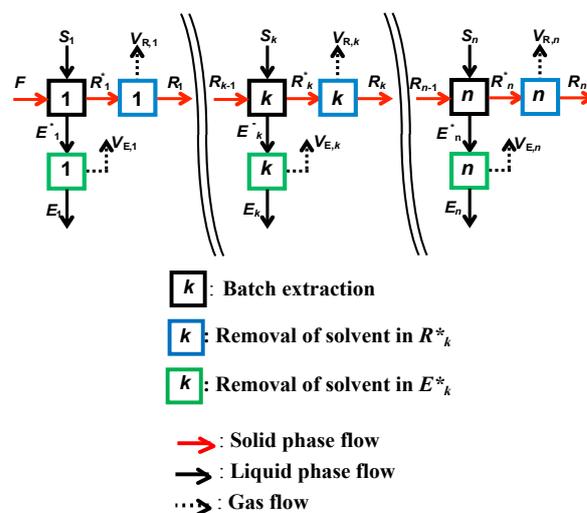
The SCG used in this study were provided in the form of frozen state from AJINOMOTO AGF, INC., and the moisture was firstly removed by leaving the sample at 383K for more than 24 hours. After removing the moisture, the pretreated SCG were used as the feed for the solvent extraction experiments. The coffee grounds from AJINOMOTO AGF, INC., were purchased and used as the virgin coffee grounds (VCG) for the solvent extraction experiments without any pretreatment.

As solvents, hexane, toluene and methanol were used. Hexane was one of the common solvents commercially used to extract food plant oils. Toluene or methanol was selected as an example of aromatic or alcohol compounds. These chemicals were in analytical grade and purchased from Fujifilm Wako Pure Chemical Co.

The procedure of multistage crosscurrent extraction by batchwise operation and experimental conditions are shown in **Figure 1** and **Table 1**, respectively. The feed coffee grounds of VCG or SCG were introduced to the 1st stage as feed,  $F$ .

The  $F$  was mixed with 100 mL solvent  $S_1$  and the mixture was shaken in isothermal bath in 24 hours, under the specified temperature. Then, the mixture was separated into the liquid  $E_1^*$  and the wet solid  $R_1^*$ . The solvent contained in  $E_1^*$  or  $R_1^*$  was removed by heating at the specified temperature and the solid  $R_1$  and extracted oil  $E_1$  were obtained.  $R_1$  was used as feed for the 2-nd extraction. The same amount of solvent  $S_k$  was added in the  $k$ -th extraction. First,

hexane was used as solvent and the extraction was carried out 10 times ( $n=10$ ). Then, the overall yield of the oil extracted from VCG and SCG were determined. Second, the solvent extractions were conducted under different extraction conditions in the cases of  $n=1$ . Hexane, methanol, toluene were used as solvents and the extraction temperature was changed at 300K, 310K and 320K. Then the influences of solvents and extraction temperature on the yield of the extracted oil were investigated.



**Figure 1** Multistage crosscurrent extraction procedure

**Table 1** Experimental conditions

Materials		
Coffee Grounds		VCG SCG
Solvent		Hexane, methanol, toluene
Extraction conditions		
Mass of sample, $F$	[kg]	0.025
Volume of solvent, $S_k$	[m <sup>3</sup> ]	$1.0 \times 10^{-4}$
Extraction time	[hr]	24
$n$	[-]	1, 10
Extraction temperature, $T$ [K]		300, 310, 320
Conditions of solvent removal in $R_k^*$ , $E_k^*$		
Temperature	[K]	353(methanol, hexane) 383(toluene)
Time	[hr]	10(hexane, methanol removal in $E_k^*$ ) 14(toluene removal in $E_k^*$ ) 48(solvent removal in $R_k^*$ )

## 3. Results and discussion

### 3.1 Definition of oil yield

The yield of the oil extracted after  $k$ -th extraction was defined as,

$$Y_k = \sum_{i=1}^k E_i / F \quad (1)$$

where,  $E_i$  and  $F$  were the oil extracted at the  $i$ -th extraction and the mass of the feed coffee grounds.

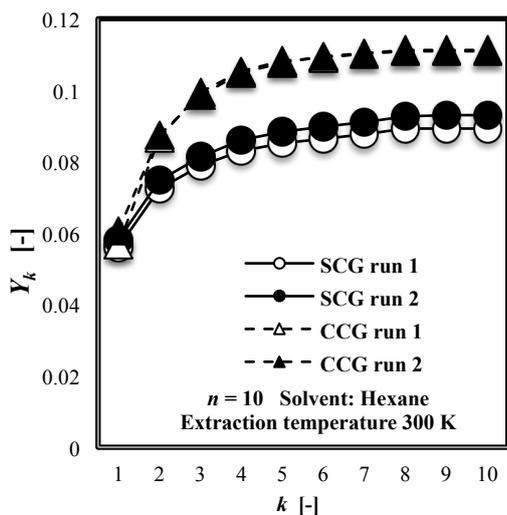
### 3.2 The yield of oil recovered from VCG and SCG

The provided SCG were dried in the atmosphere at 383K and the mass became constant after more than 24 hours. The ratio of the dried mass to the initial wet mass was obtained as 0.26 kg-dried/kg-wet and the SCG pretreated for more than 24 hours were used as the extraction feed in all cases.

**Figure 2** shows the plots of  $Y_k$  against the stage number of extraction  $k$  with VCG and SCG. In the case of VCG, the solvent extraction was conducted twice.  $Y_k$  increased gradually from 0.056 to 0.111 while  $k$  ranged from 1 to 8 and had nearly no change with  $k > 8$ .  $Y_{10}$  was 0.111 while the yield of oil extracted at first time  $Y_1$  was more than 0.058, it meant 0.52 of total oil was recovered at the first time extraction. In the case of SCG, the solvent extraction was also conducted twice and their results were mostly the same.  $Y_k$  increased gradually from 0.056 to 0.093 (run 2) or 0.089 (run 1) while  $k$  ranged from 1 to 8 and had nearly no change with  $k > 8$ .  $Y_{10}$  ranged between 0.089 and 0.093 while the yield of oil extracted at first time  $Y_1$  was more than 0.056, it meant more than 0.60 of total oil was recovered at the first time extraction.

The total recovered oil of VCG is 0.111 higher than that obtained of SCG (0.093). Although the SCG were not obtained from the VCG used in this study and the reason was unclear, some parts of oil might have been taken at the brewing step.

$Y_k$  of VCG and  $Y_k$  of SCG were mostly the same at  $k=1$  and different with  $k \geq 2$ . Although the solubility of oil in hexane was not determined in this study, at the first time extraction ( $k=1$ ) the solubility limit of oil in hexane might have been exceeded.

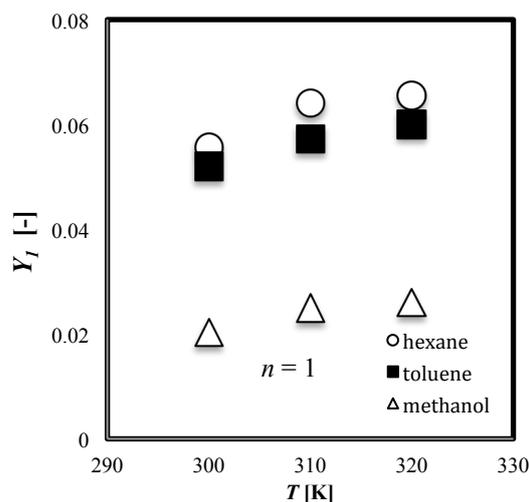


**Figure 2** Yield of oil extracted from VCG and SCG

### 3.3 Influences of solvents and extraction temperature on the yield of oil

**Figure 3** shows the influences of solvents and extraction temperature on the yield of oil  $Y_1$ . Hexane

showed the highest  $Y_1$  followed by toluene and methanol.  $Y_1$  in the cases of hexane and toluene were comparable and  $Y_1$  in the case of methanol was much smaller. This trend was also reported in previous study [3]. The main components of oil must have been triglycerides of free fatty acid C16 or C18 therefore they were soluble more in non-polar solvent. The yield of oil  $Y_1$  increased slightly as temperature increased. Although the effects were insignificant in this measurement range, the solubility increased as the temperature increased.



**Figure 3** Influences of solvents and extraction temperature on the yield of oil

## 4. Conclusions

From the experiment on removing moisture from the provided SCG, the ratio of the dried mass to the initial wet mass was obtained as 0.26 kg-dried/kg-wet. From the experiment on determining the overall yield of the oil recovered from VCG and SCG,  $Y_{10}$  of VCG was 0.111,  $Y_{10}$  of SCG ranged between 0.089 and 0.093. From the experiment on investigating influences of solvents and extraction temperature on the yield of oil, hexane showed the highest  $Y_1$  followed by toluene and methanol.  $Y_1$  increased slightly as extraction temperature increased.

## 5. References

- [1] Angellina., "Coffee, the second most popular drink in the world" , Eastwest, 2016 January.
- [2] T. Tokimoto et al., "Removal of lead ions in drinking water by coffee grounds as vegetable biomass", Journal of Colloid and Interface Science, 281, 56-61, 2005.
- [3] Ioannis et al., "Influence of solvent selection and extraction temperature on yield and composition of lipids extracted from spent coffee grounds", Industrial Crops & Products, 119, 49–56, 2018.