Effect of Moisture and Particle Size on the Extractability of Oils from Seeds with Supercritical CO$_2$

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ABSTRACT

Moisture level and particle size of soybeans, peanuts and cottonseed were correlated with the extraction rate and yield of oil when extracted with supercritical carbon dioxide (SC-CO$_2$) at a constant temperature (50 C) and pressure (8000 psig). The rate of extraction and ultimate oil yields were quite low with cracked soybeans. However, good extraction rates and nearly theoretical oil yields were obtained from ground or thinly flaked (<0.010") seeds. Moisture levels between 3% and 12% had little effect on extractability. Oil composition was not influenced by either parameter. Scanning electron microscopy was used to study seed structure before and after extraction with SC-CO$_2$. Micrographs of SC-CO$_2$ extracted seeds were similar to hexane-extracted seeds.

INTRODUCTION

German scientists have investigated the extraction of natural products using supercritical fluids since the early 1960's and have demonstrated that carbon dioxide above its critical temperature and pressure is a suitable solvent for the extraction of oil (1-4). Subsequently, Friedrich and co-workers have reported high soybean oil recovery with supercritical carbon dioxide (SC-CO$_2$). Phosphorus and iron contents of the SC-CO$_2$-extracted oil were lower and the oil color was lighter than characteristic of hexane-extracted crude oil. Flavor scores of the refined soybean oil extracted by SC-CO$_2$ were not different from refined hexane-extracted oils (5). Stahl et al. showed that oil yield was dependent on particle size and structure of the oilseed (6). The present study shows the effect of particle size and moisture content of the seed on the extractability of oil with SC-CO$_2$. The scanning electron microscope (SEM) was used to determine how the native structures of the oilseeds change during extraction and to ascertain the best configuration of the seed for efficient oil extraction with SC-CO$_2$.

EXPERIMENTAL

Whole soybeans were dried or tempered to 3.5%, 6% and 12% moisture levels which were determined gravimetrically using a Brabender oven; the soybeans were then cracked and dehulled. One-third of the cracked beans were flaked to a thickness of 0.25 mm, another third were ground to a fine flour (over 94% of the flour passed through a 100 mesh screen, which corresponds to 150 microns) by an Alpine Mill, and a final third were used without flaking or grinding. The cracked, ground or flaked soybeans (950 g) were

REFERENCES


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placed into a 2-l extractor as described previously (5). SC-CO$_2$ extractions were conducted at 8000 psig and 50 C. Extracted oil was removed from the receiver every hour and weighed. The extraction was terminated when the oil recovered per hr dropped to less than 1 g.

In a second study, cracked, dehulled soybeans were flaked to average thicknesses of 0.10, 0.25, 0.38 and 0.81 mm and were exhaustively extracted until less than 0.25 g of oil was obtained during the last one hr of extraction. An additional experiment was conducted to determine whether soy flakes or soy flour was extracted more readily. In this case, 46 grams each of flour (> 150 μ) and flakes (0.10 mm) were separately extracted in a 150-ml vessel under identical conditions of 8000 psig and 50 C. Extracted oil was weighed after each of 5 successive 50 standard liter aliquots of CO$_2$ had passed through the column.

Cottonseed and peanuts were cracked, dehulled and flaked before extraction; neither could be ground because of their high oil content. Extraction conditions were 8000 psig at 50 C for cottonseed and 10,000 psig at 70 C for peanuts.

Residual oil in the extracted oilseed samples was determined by AOCS Official Method Ac 3-44 (7). Extracted samples were analyzed for free fatty acids and unsaponifiable material by AOCS Official Methods Ca 5a-40 and Ca 6a-40 (7).

Methyl esters were prepared from the oil and analyzed by gas liquid chromatography (GLC) to determine fatty acid composition.

Samples for determination of surface structures of the different particle configurations were prepared for SEM examination by established methods (8).

RESULTS AND DISCUSSION

Either flaking or grinding prior to SC-CO$_2$ extraction, so that the surface area increased or a greater number of cell walls ruptured, was necessary for good oil recovery (Table I). These results agreed with those of Othmer and Agarwal who showed that soybeans had to be flaked or ground to permit the extraction of oil by hexane (9). After extraction with SC-CO$_2$, residual oil in cracked soybeans was about 20%, whereas 2% or less oil remained in the flaked and ground samples. Transfer of oil through the cell walls of the cracked beans did not occur, and only the surface oil that was exposed by the minor fracturing of the cell walls during the cracking process was removed. More cell walls were broken in the further process of flaking or grinding, resulting in a greater oil yield. Extractability of oil from flaked and ground beans was improved considerably over that obtained from the cracked beans. Oil solubility reached the apparent equilibrium solubility of 2.5% for our extraction conditions as indicated by the constant rate of extraction (straight portion of the extraction curves in Fig. 1). We found that moisture levels within the range of 3-12% had little effect on the extractability of the 3 different soybean structures. Moisture content also did not affect the extractability of the oil from dry-milled corn (10) or lupine seed (11). Oil is more soluble than water in SC-CO$_2$; therefore oil was removed first. Moisture content of the extracted meal was greater than full fat meal by an amount somewhat less than that due to oil removal alone. After extraction, the meal was examined. The moisture content increased steadily through the extractor in the direction of SC-CO$_2$. Extracted water is minimal and is observed only during the

**TABLE I**

<table>
<thead>
<tr>
<th>Particle configuration</th>
<th>Moisture (%)</th>
<th>Res. oil (%)</th>
<th>Oil solubility (%)</th>
<th>Unsp. (%)</th>
<th>FFA (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cracked</td>
<td>3.5</td>
<td>20.8</td>
<td>0.46</td>
<td>0.94</td>
<td>0.44</td>
</tr>
<tr>
<td></td>
<td>6.0</td>
<td>18.4</td>
<td>0.39</td>
<td>0.81</td>
<td>0.42</td>
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<tr>
<td></td>
<td>12.0</td>
<td>20.3</td>
<td>0.43</td>
<td>1.08</td>
<td>0.44</td>
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<tr>
<td>Flaked (0.25 mm thick)</td>
<td>3.5</td>
<td>2.1</td>
<td>2.26</td>
<td>0.63</td>
<td>0.35</td>
</tr>
<tr>
<td></td>
<td>6.0</td>
<td>0.9</td>
<td>2.76</td>
<td>0.70</td>
<td>0.34</td>
</tr>
<tr>
<td></td>
<td>12.0</td>
<td>1.1</td>
<td>2.54</td>
<td>0.65</td>
<td>0.44</td>
</tr>
<tr>
<td>Ground (94% &lt;100 mesh)</td>
<td>3.5</td>
<td>0.9</td>
<td>2.30</td>
<td>0.83</td>
<td>0.28</td>
</tr>
<tr>
<td></td>
<td>6.0</td>
<td>0.7</td>
<td>2.40</td>
<td>0.74</td>
<td>0.27</td>
</tr>
<tr>
<td></td>
<td>12.0</td>
<td>1.8</td>
<td>2.38</td>
<td>0.74</td>
<td>0.36</td>
</tr>
</tbody>
</table>

*a* Determined by AOCS Official Methods.

*b* Apparent solubility determined by oil wt./CO$_2$ wt.