Water Accumulation in the Alcohol Extraction of Cottonseed

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The critical moisture content of cottonseed flakes extracted with an aqueous alcohol solvent can be defined as that flake moisture level at which the flakes lose no moisture during extraction. This study shows that the critical moisture content for aqueous ethanol (92%, w/w) is 3%. For aqueous isopropanol (88%, w/w) this value is 6%. If the moisture contents of the flakes are above these levels, then the solvents pick up moisture. For moisture contents below this level, the flakes adsorb moisture and actually dry the alcohol. It is proposed that this latter capability can be used as a basis for a method to control water accumulation in aqueous alcohol solvent extractions.

KEY WORDS: Alcohol, cottonseed, extraction, oilseeds, water.

For over ten years there has been a concerted interest within the oilseed crushing industry in the development of an alternate solvent to replace hexane for the extraction of vegetable oil (1,2). This was initially spurred on by the petroleum crisis of the late seventies but was later fueled by interest in finding a solvent that was less flammable and thus safer to use and that would produce less noxious emissions. Another potential benefit of a new solvent to the cottonseed industry would be the removal of gossypol and any aflatoxin that might be present from the extracted meal. Aqueous ethanol and aqueous isopropanol currently appear to be the most likely candidates for such a solvent. They are biorenewable, much less flammable than hexane and have the ability to remove gossypol and aflatoxin.

Extensive knowledge of equilibrium data is required for the development of any chemical process. Data relating the amounts of oil, solvent and flakes for a solvent extraction with either aqueous ethanol or aqueous isopropanol have been reported (3). Work has been done in the past to determine the solubilities of vegetable oils in various ethanol-water and isopropanol-water mixtures (4–6). These studies have all shown that addition of moisture to alcohol significantly reduces its oil solubility. For example, near its boiling point, 78°C, ethanol is totally miscible with cottonseed oil when the alcohol's water content is less than 2% (w/w). A water content of 5%, *i.e.* 95% (w/w), reduces the solubility of oil in the ethanol to below 20%. The azeotrope of ethanol (92%, vol/vol) has an oil solubility of 10%. Similar results are shown for isopropanol, although its miscibility with oil is somewhat greater than that of ethanol at a given water concentration. It should be pointed out that the increase in solubility of the absolute alcohols does not make them attractive solvents for extraction due to the difficulty of removing all accumulated water. It is for this reason that aqueous ethanol and isopropanol near their aceotrope have become the alternate solvents of choice.

Others have shown (7,8) that if the moisture level in flakes was below 3%, aqueous ethanol was an effective extraction solvent. One can postulate that the alcohol solvents remove and accumulate the oilseeds' moisture during extraction. As discussed above, this additional moisture decreases oil solubility and, in turn, decreases extraction efficiency. This study seeks to investigate the significance of this critical moisture level and to suggest process modifications that will keep the recycle solvent's moisture content below this amount.

MATERIALS AND METHODS

The mill-run cottonseed (dehulled seed with hulls to give 41% protein and donated by the Yazoo Valley Oil Mill, Greenwood, MS) was produced in the Mississippi Valley region from the 1990 crop. The aqueous ethanol was commercial-grade and met U.S.I. specifications for 190 proof (92% w/w). The aqueous isopropanol was made from reagent-grade isopropanol (99.9% w/w) diluted to its azeotrope of 87.7% (w/w). The solvent moisture contents were checked with a Brinkman 701 Karl Fischer (K-F) titrator (Karlsruhe, Germany).

In a typical experiment, enough water was sprayed on the seed to result in a final moisture level of about 14%, mixed in a batch mixer and put in a closed container for about 12 h to allow the moisture to evenly distribute. The meats were then flaked and put into a forced draft oven, in 450 g batches, at 130°C. The flakes were dried for times varying between 5 and 120 min. At various time intervals, they were removed from the oven and charged into a bench-top extractor (3). A small sample of flakes at each drying time used was set aside for gravimetric moisture determination. About 600 g of solvent was circulated through the extractor at the solvent's boiling point for 2 h. The actual moisture content of the solvent entering the extractor for each run was determined with the K-F titrator. After extraction, the miscella was drained and also analyzed for moisture with the K-F titrator. The amount of oil in the miscella was determined by removing the water and solvent with a rotary evaporator. The amount of water in the solvent after extraction can then be calculated.

RESULTS AND DISCUSSIONS

Figure 1 shows that, for drying times varying between 5 and 120 min, moisture varies between 13 and 2%. Note that no attempt was made to optimize the drying procedure, and a more efficient method would decrease the time required to reach a given moisture level. However, the results do show that it is relatively easy to reach a 4% moisture level and difficult to dry below 2%.

Figure 2 is a plot of percent moisture in the flakes entering the extractor *vs.* the change in solvent moisture after extraction when aqueous ethanol (92% w/w) is used as the solvent. This change is calculated by subtracting the percentage moisture in the feed solvent from the percentage moisture in the solvent after extraction. For example, if the flakes had a moisture level of about 7% entering the extractor, the ethanol would go from 92% (w/w) purity to 90% (w/w). This, as already discussed, cannot be allowed to happen. If the flakes are dried to around 3%, there is no accumulation of water. This value is in agreement with

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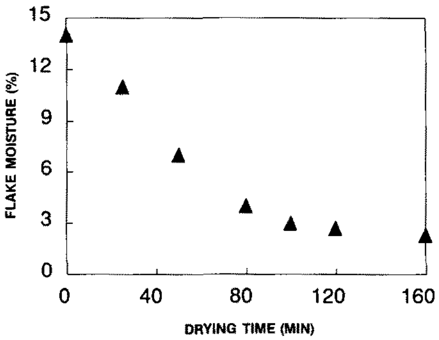

FIG. 1. Flake moisture *vs.* drying time in a laboratory convection oven at 130°C.

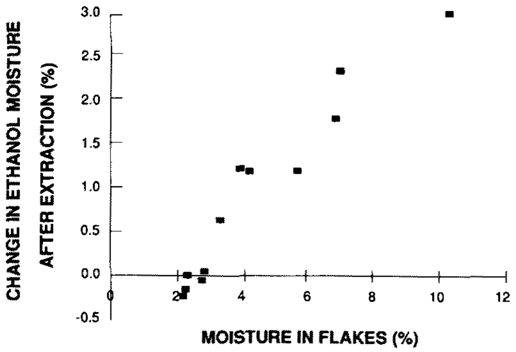

FIG. 2. Change in ethanol moisture after oil extraction from flakes with various initial moisture levels.

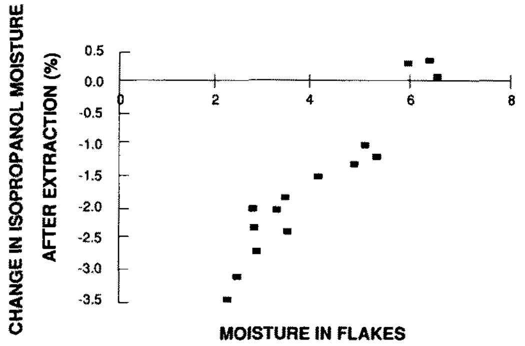

FIG. 3. Change in isopropanol moisture after oil extraction from flakes with various initial moisture levels.

previous studies (7,8) that found a significant reduction in the ability of aqueous ethanol to extract oil from flakes with a moisture level greater than 3%. Note that if the flakes are dried to below 3%, water is actually removed from the solvent. This suggests a potential method to dry aqueous alcohol and has been proposed as a method to produce absolute ethanol with corn as the drying medium (9). This point is explored further below.

Figure 3 shows the change in moisture of the solvent *vs.* initial moisture of flakes when aqueous isopropanol is the extraction solvent. The equilibrium moisture level for isopropanol is apparently higher than that of ethanol. The flakes need to be dried to only around 6% to achieve no water accumulation in the solvent. Note that solvent drying also occurs at flake moisture levels below 6%.

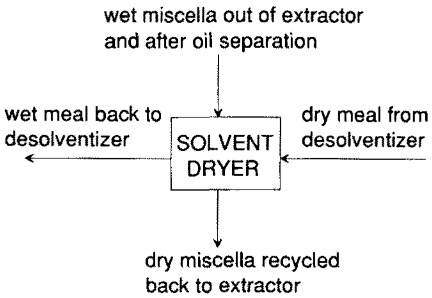

FIG. 4. Proposed scheme for controlling alcohol moisture during oilseed extraction.

It should be pointed out that the solvent-to-feed ratio used in this study is much lower than would be used in an actual extraction process. However, the solvent-to-feed ratio has no effect on the equilibrium moisture levels. The solvent-to-feed ratio only affects the number of times the ethanol can be recycled before it reaches equilibrium with the flakes. For a given moisture level of flakes coming into an extractor, the alcohol will eventually reach an equilibrium moisture value, which depends on the flake moisture and will not vary for different solvent-to-feed ratios.

After dehulling and prior to other preparation steps, cottonseed has about 8% moisture. If this seed is extracted in an alcohol process, water would accumulate in the solvent whether ethanol or isopropanol were used. To prevent this water accumulation, some type of drying operation is needed. One solution is to dry the flakes to below their critical moisture level. This may not be appropriate, however, if this drying changes the characteristics of the flakes and interferes with gossypol and aflatoxin removal. Whether this occurs is the subject of current research. Another solution is proposed in Figure 4. Here, accumulated moisture is removed from the miscella after extraction and oil separation. This is done by passing it through a bed of dry meal coming out of the desolventizer. The wet meal is sent back to the desolventizer, and the dry miscella is recycled back to the extractor. The feasibility of this proposal in an actual extraction process is being investigated.

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